

MONTHLY PROGRESS REPORTS

Period Covered
February 1 - 28, 1983

2022203075

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RESEARCH CENTER
RICHMOND, VIRGINIA

MONTHLY PROGRESS REPORTS

Period Covered
February 1 - 28, 1983

Date Issued

March 15, 1983

2022203076

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2022203077

CHARGE NUMBER: Various

DATE: March 8, 1983

PROGRAM TITLE: Analytical Research

WRITTEN BY: G. Vilcins, G. H. Bokelman, W. N. Einolf, B. W. Good,
B. M. Handy, W. R. Harvey, C. S. Kroustalis, and W. R. Morgan.

I. TOBACCO

A. Tobacco Accountability

A study is being coordinated by Robert Mullins in the MC to test the validity of using calcium, magnesium or potassium as a tracer for material accountability. The aftercut spray application point was selected for this pilot study based on negligible apparent volatile and material loss. Composite sampling was performed, mid-run, before and after the totalizer belt where cut filler, E-S or ET and spray are combined. Accountability percentages using weigh belt, OV and cation determinations will be made. An experimental design has been constructed and samples have been received.

B. Tobacco Headspace

TENAX^R Adsorbed Components

Using discriminant and factor analysis, definite components were found to be related to the presence of bright, burley and oriental tobacco. While peaks were also found that distinguished cased from uncased samples, the latter's peaks were always higher.

Using stepwise regression analysis, and 4 blends of bright and burley, peaks were selected to predict blend composition of bright and burley. In all cases the important peaks were larger in burley. The precision of the predicted composition was $\pm 4\%$.

C. Sulfate in Tobacco

Ashing was found to be necessary for the sulfate determination due to an unidentified interference when the unwashed sample was extracted. A cation ion exchange resin in the silver form was found to be invaluable in removing the chloride from the HCl extract of the ash prior to ion chromatography. Precision was found to be $\pm 2\%$ at the 2-Sigma level for this determination.

D. Isolation and Examination of Tobacco Cell Wall Components

The E-55 bright tobacco residue used previously in extraction of "glycoprotein" with two sequential treatments of mild HOAc-NaClO₂ solutions was given a third treatment using more severe conditions. This last treatment was successful in removing more of the "glycoprotein." The filtered, dialyzed and lyophilized extract was found to contain 8.51% total nitrogen, which suggests that its protein content is in excess of 50%.

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E. Mass Spectrometry

An analysis of South American and Mexican Winston and Marlboro cigarettes for sucrose esters showed that no synthetic sucrose esters could be detected in the Winston cigarettes. A comparison of flue-cured and air-cured bright tobaccos showed that all sucrose esters were present at a higher level in the air-cured sample than in the flue-cured. When a correction was made based on the weight loss of the air-cured tobacco (essentially placing the results on a per-leaf or leaf-area basis) all but 25% of the difference in ester content could be accounted for. Additional green tobacco callus was analyzed for sucrose esters. Two growth experiments, Gibco and YIIIM, showed the highest ester levels, but these levels were far below those found in mature tobacco leaves.

II. FLAVOR

A. CF

Twenty-seven domestic and two international brands were analyzed for CF. Of the domestic manufacturers only R.J. Reynolds uses CF in their low delivery products. Reconstituted tobacco from Vantage showed no CF concentration differences from the rest of the filler indicating that CF is sprayed on filler.

B. Sucrose in CTB

Sucrose, glucose and fructose levels are being determined in samples taken from various points in the process. Samples include CT, burley, burley spray and CT slurry (burley spray plus CT). The GC and LC procedures are both being used for this study to determine the more appropriate technique. There is evidence of hydrolysis of the sucrose during the LC extraction and cleanup that is not evident in the GC extraction (70% methyl alcohol). Recoveries of sucrose are ~83% with LC versus 95% by GC. Recoveries are based on a laboratory formulation of burley spray with CT added. The purpose of this manufacturing scale experiment is to determine the fate of sucrose during spray formulation, application to tobacco and drying.

C. Room Aroma Evaluation

The descriptors for 10 full flavored burley and oriental cigarettes were evaluated using BMDP7D and BMDP2V for ANOVA. Two conclusions were drawn: 1) that burley and oriental are significantly different from each other and 2) that all tested full flavored cigarettes are very similar and show few distinguishing characteristics.

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III. SMOKE

A. Atomic Absorption Studies

Work continued on the study of the effects of filtration and dilution on the delivery of cadmium, nickel, chromium, and cobalt into mainstream smoke. Most of the work centered around the development of smoke collection procedures and analysis methods.

B. Nitric Oxide in Sidestream Smoke

The dynamic and static burning coal temperatures, the amounts of soluble ammonia and total alkaloids were obtained for the series of cigarettes used in the sidestream smoke NO study. A correlation existed between dynamic burning coal temperature and total alkaloids with nitric oxide in sidestream smoke. No significant relationship was obtained between NO in sidestream smoke and static burning coal temperature, soluble ammonia, and protein (estimated value). Several observations were made from the additional results:

- 1) Bright tobacco cigarettes with low nitrate content and low NO in mainstream delivery had higher dynamic burning coal temperature than high nitrate content tobacco cigarettes.
- 2) Expanded bright, burley and oriental had higher dynamic burning coal temperature than unexpanded bright, burley, and oriental tobacco cigarettes.
- 3) Static burning cigarette coal temperatures were not significantly different for the cigarettes studied.
- 4) Dynamic burning coal temperature was about 300°C higher than the static burning coal temperature.
- 5) Although, no changes were observed in the values of total nitrogen after the expansion process, the relative value for total alkaloids decreased and the relative estimated protein value increased.

IV. NMR SPECTROMETRY

A. Serricornin

In a joint effort with D. Williams, we have started a program to understand the stereochemistry of the compounds involved in the synthesis of the tobacco beetle sex pheromone, serricornin. The major goals of this program are the correlation of stereochemistry with chromatographic behavior and to insure that a consistent product is given to the warehouse people for testing the efficiency of the serricornin as a trapping agent.

James V. Williams

2022203080

CHARGE NUMBER: 0307
PROJECT TITLE: Measurements Development
PROJECT LEADER: C. L. Irving
PERIOD COVERED: February, 1983
DATE OF REPORT: March 9, 1983

I. Loose Ends Measurement

Objective: To develop a dynamic sensor and/or laboratory instrument for measuring loose ends and determine the effect of particle size, particle mixing, blend formula, tobacco weight, and OV on cigarette loose ends.

Status: A test was completed to evaluate the viability of using the Borgwaldt Cigarette Ends Tester or PM Loose Ends Tester for measuring cigarette loose ends as well as to determine the effect of DIET level, DIET shred size, tobacco weight, and processing OV on loose end generation. For the test, Cambridge type blends were made with 10, 20 and 30% normal production DIET, as well as, with 30% "gentle" tower DIET, 30% > 12 mesh DIET, and 30% < 12 mesh DIET. Results of the study showed shred size and processing OV, which affects shred size, to be the major factors affecting loose ends. At constant shred size increasing tobacco weight reduced loose ends as expected. The test results gave no clear indication of the effect of DIET level on loose ends. The results showed however, that for DIET a trade off does exist between filling power and shred size and that for DIET the shred size is the major factor affecting loose ends not filling power.

The test results showed the Borgwaldt device to be fundamentally sound and to produce results that followed logical and expected patterns. The PM tester, with the conical probe, gave results which exhibited trends similar to the Borgwaldt but with some sensitivity to filling power. The approach used in the PM tester, however, does appear viable for quantifying loose ends dynamically.

Plans: A similar test will be repeated to verify the effects and interactions of shred size, OV, tobacco weight, and DIET level on loose ends. In addition, different size cones will be evaluated on the PM unit to reduce the sensitivity to tobacco filling power. Testing will also be done with typical production cigarettes to develop a baseline of data for comparison to experimental results.

II. Dynamic Firmness

Objective: To develop an instantaneous non-destructive technique for determining the firmness of cigarettes during making.

Status: A test was completed in the R&D Semiworks to evaluate the effect of tobacco temperature on the variability of dynamic firmness results. For the test, Marlboro filler was sent to the maker at 43, 77, and 88°F and subsequently exited the garniture at 60, 83, 90°F, respectively. Analysis of the data showed tobacco temperature to be a significant variable for accurately predicting compacimetric firmness from transducer data. Tobacco temperature caused ≈ 0.2 firmness units per°F offset in predicted firmness results.

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Plans: An Apple minicomputer with an Isaac interface was ordered to evaluate the entire dynamic firmness control concept (with dynamic moisture). Testing of this system will begin after the necessary interfacing hardware to move the ecreteur blade is obtained. In addition, a test will be run to evaluate the effect of tobacco shred size on the precision of dynamic firmness results.

III. Bulk Tobacco Filling Power

Objective: To develop instrumentation for more quickly determining the filling power of cut filler.

Status: Two tests of the four Borgwaldt CV testers were completed. The first test, using Marlboro and DIET tobaccos at three OV levels each, showed some indication of differences (probability level of 93%) between the original Borgwaldt CV tester and the three new units. After some mechanical adjustments the second test, with Marlboro, DIET, RL, and burley at three OV levels each, showed no evidence of instrument to instrument variation.

Plans: Make a recommendation to management on the use of the Borgwaldt CV tester as a replacement for the existing CV test.

Christopher L. Gray

2022203082

CHARGE NUMBER: 0400
PROJECT TITLE: Tobacco Properties Applications
PROJECT LEADER: J. F. Sherwood
PERIOD COVERED: February, 1983
DATE OF REPORT: March 8, 1983

I. Whole Leaf Cutting

Objective: Develop a process for cutting and processing whole leaf tobacco into cigarette filler.

Status: An experiment was initiated to characterize the size and filling power properties of a single grade of bright filler (E4X) produced by the two whole leaf cutting approaches under development: rolling and cutting, and cutting followed by air separation of stem. Approximately 400 lbs of E4X bright leaf has been processed through Engineering's leaf opening pilot plant at the Stemmy and rolled using the Quester stem roller in D Pilot Plant. Cutting and drying of the leaf in the Semiworks is scheduled for this week. Also, approximately 1200 lbs of the E4X leaf was threshed at a Universal Leaf subsidiary to obtain control material. As soon as PM USA Engineering completes modifications to the small Witte separator (1-2 weeks) to improve its performance, a third portion of the E4X will be cut and processed through the D&F feeder and Witte separator to remove the stem.

Plans: Complete filler and cigarette analyses on the three bright samples. Move the Quester stem roller to the Stemmy and conduct studies to optimize leaf opening and rolling parameters.

II. Tipped Burley

Objective: Evaluate the filler size and filling power of burley filler produced from tipped burley farmer bales.

Status: Strip size and coal strength analyses were completed on samples produced from tipped burley test tobacco and from control threshed burley. As with the filler CV and sieve results, the test samples showed directional but not statistically significant improvements over the controls. Firmness and filler stem content analyses were completed and the data are being analyzed.

Plans: Complete data analyses and issue a summary report.

III. Wavy Cutting

Objective: Evaluate AMF-Legg's wavy cutting process to determine any improvements on cigarette properties.

Status: A test was completed comparing the filler and cigarette characteristics of DBC bright and RL-TC tobacco cut conventionally and with AMF-Legg's wavy cutting process. No improvements in filling power or cigarette coal strength were

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observed with the wavy cut tobacco.

Plans:

AMF-Legg is planning to conduct tests to investigate the effect of the shred wavelength, amplitude, and cut width on cut filler. If AMF-Legg finds improvements, we may re-evaluate the process.


J. F. Sherwood

/nb

CHARGE NUMBER: 1005
PROJECT TITLE: Improved Semiworks Operation
PROJECT LEADER: R. T. Gaudlitz
PERIOD COVERED: February, 1983
DATE OF REPORT: March 4, 1983

Title: Hauri Steam Tunnel (T. Skidmore)

Objective: Develop a process which uses the superwetting of cut filler and the co-current Adt dryer to achieve the maximum CV exiting the dryer.

Status: The steam tunnel has been installed ahead of the Semiworks Adt dryer along with a conveyor system which allows filler to enter the dryer via the tunnel or by by-passing the tunnel. Some additional work is necessary in duct modifications, instrumentation, and steam and water connections.

Plans: The tentative schedule is:

March	: Complete Installation
April - May	: Preliminary Tests
May - July	: Final Evaluation

Title: Variable Angle Doffers - R&D Semiworks (T. Skidmore)

Objective: Minimize tobacco breakage during silo discharge by optimizing silo doffer angle and design.

Status: Per a joint recommendation by R&D and Manufacturing Engineering, pin doffers will be installed on cut filler silo No. 12 in the MC during the March 4-6 weekend and evaluated by Manufacturing Engineering on an MFKS blend.

Pin doffers were used to discharge an MJL blend (which has a higher percentage of ET than Marlboro) from the Semiworks final blend silo. This material discharged without problems. A 1.9% loss of longs, across the silo, did occur. This is a greater loss than the < 1.0% experienced with MFKS.

A work order has been issued to modify the silo doffer for evaluation with bright strip.

Plans: A new doffer design and the effect of doffer rpm on strip damage will be examined.

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Title: Adt Dryer Water Spray (R. Wagoner)

Objective: Evaluate a water spray system in the Adt dryer as a means of achieving CV improvement.

Status: Two sets of test runs were made in which Marlboro filler cut at 20.4 and 21.9% OV values was dried in the Semiworks Adt dryer that is equipped with three water spray nozzles located in the feed end. During the first set (20.4% filler) additional water was sprayed on the filler at rates that would translate into moisture gains of 1.1, 2.8, and 4.5%. Water rates producing theoretical gains of 1.5 and 3.2% were sprayed in the second set (21.9% filler). Control tests with no water spray were also run for each set. Cylinder volume results showed no CCV gains for either control run, while the test runs produced gains of 1.5 - 1.9 cc/10g. Analysis of the sieve data showed an approximate 2% loss in longs + mediums across the dryer for the control and test runs.

Plans: 1) Incorporate any further testing into the Hauni steam tunnel program.
2) Assist Manufacturing should they decide to test a water spray system as part of their CV enhancement program.

Title: Semiworks Witte Cooler and Separator Unit (R. Wagoner)

Objective: To evaluate the Witte fluidized bed equipment, located in the Semiworks, as a separator.

Status: In a co-operative effort with Process Engineering, modifications to their smaller Witte unit are being made that will permit the hood and chimney ratios to be varied. Also, during tests the internal bags will be removed to closer simulate the larger separator. This decision to modify the smaller unit was made to minimize the cost and time required by the overall evaluation program.

Plans: 1) Run joint tests with Engineering on the modified unit to determine the optimum design specifications for the larger Witte unit.
2) Evaluate bags constructed from mesh sizing cloth as a possible dust removal system for a Witte unit having an internal baghouse.

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Title: TOP and Dickinson Conditioning Units (R. Wagoner)

Objective: Test the performance of the TOP and Dickinson conditioning systems in comparison with the PM vacuum conditioning system.

Status: A demonstration run was made for representatives of Rothmans conditioning Canadian bright cases in the TOP unit. Following this, and acting upon their recommendation to do partial conditioning in the ordering cylinder, a comparison run among the TOP, Dickinson and vacuum conditioner was made with Canadian bright cases. Two different conditions were tested on the TOP system: 1) with desuperheater water addition and a "cold" ordering cylinder, 2) without desuperheater water and a "hot" ordering cylinder.

Plans: Evaluate the moisture and strip sieve samples collected during the test. Also, cut a composite sample of the Canadian bright to examine the effects on filling power.

Title: Semiworks Instrumentation & Computers (R. Shimaitis)

Objective: Implement supervisory control, direct control and reporting of the primary process.

Status: Power: The firmware expansion module board was replaced and the computer is still failing. This indicates that the original FEM board hardware is functioning properly, however, the Fisher supplied microcode is still questionable. The Cabarrus Primary computer is experiencing similar computer failures.

Software: The moisture and weighbelt calibration software user interface needs to be cleaned up. It is awkward and the 11 second response time is unacceptable. Rework is in progress. The new graphics plotter has been added to the system. Software has been completed to easily plot overhead transparencies. The Hauni Steam Tunnel has been configured into the system, and added to the data acquisition software.

Hardware: The Hauni Steam Tunnel has been installed. Controller and sensor wiring is in progress.

Plans: 1) Installation of the Motor-Generator Set and the Power Distribution System is to begin on March 14.
2) Complete control algorithm software for Hauni Steam Tunnel.
3) Cleanup calibration software.

Robert T. Gandberg

2022203087

CHARGE NUMBER: 1101
PROGRAM TITLE: ENTOMOLOGICAL RESEARCH
PROJECT LEADER: J. S. Long
PERIOD COVERED: February 1-28, 1983
DATE OF REPORT: March 7, 1983

I. CIGARETTE BEETLE PHYSIOLOGICAL STUDIES

A. Sugar Alkaloid Study

The second replication of the study evaluating the effects of sugar and alkaloid content on beetle growth and development is nearing completion. Earlier data is being confirmed. Additionally, the effects on fertility and fecundity are under study.¹

B. Cold Temperature Study

1. Laboratory Study

A test has been initiated to determine the length of time necessary to effect 100% mortality of all life stages of the cigarette beetle at -3.9°C.²

2. Low Temperature Fumigation with Magnesium Phosphide

Three studies were completed. One hundred percent mortality was achieved under tarpaulin, in a container/van, and in a warehouse. Temperature ranges were from 46-49°F. Dosages ranged from 20 g/1000 ft³ in the warehouse and under tarpaulin to 33 g/1000 ft³ in the container. The TLV (0.3 ppm) was reached in 48 hours after aeration except in tobacco enclosed in polyethylene which required three weeks to reach the TLV.³

C. Larval Symbiont

Pastry flour will serve as a nonnutritive diet for studying the role of the mid-gut symbiont in development.⁴

D. Antifeedants

Three additional antifeedant candidates have been received for evaluation. All three are constituents of citrus pulp.⁵

E. Pupal Cells

Hexane extraction does not remove the toxic agents from pupal cells. Extracted cells, mixed with bright tobacco (30% concentration) yielded 90% mortality. It appears that a MeOH extraction route contains some toxic material.⁶

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II. ASSISTANCE TO OTHERS

A. Dock Street, Stockton Street, and 20th Street

Efficacy tests were conducted at Dock Street, Stockton Street and 20th Street during the month.^{7,8,9}

B. Liners

A light weight Tyvek® liner and a 50 lb. weight Kraft paper liner have been received for evaluation as possible replacements for regular Tyvek® liners.¹⁰

III. REFERENCES

1. M. F. Minor. Notebook No. 7197, pp. 128-129.
2. S. Drew. Notebook No. 7850, p. 21.
3. D. L. Faustini. Notebook No. 7746, pp. 69-73, 79.
4. R. M. Lehman. Notebook No. 7721, p. 111.
5. J. S. Long. Notebook No. 7777, p. 10.
6. D. L. Faustini. Notebook No. 7746, pp. 68, 81-82.
7. D. L. Faustini. Notebook No. 7746, p. 78.
8. R. M. Lehman. Notebook No. 7721, pp. 112-113.
9. S. Drew. Notebook No. 7850, p. 20.
10. J. S. Long. Notebook No. 7777, p. 12.

nwp



2022203089

CHARGE NUMBER : 1307

PROJECT TITLE : Reconstituted Tobacco Development

PERIOD COVERED: February 1-28, 1983

PROJECT LEADER: G. Gellatly

I. APPLICATION OF CLASS TOBACCOS TO TOBACCO MATERIALS

A. Objective

To define processes for the application of class tobaccos to tobacco materials and to define the physical and subjective character of the resulting products.

B. Status

1. The pilot plant products required to evaluate the effect of RL coating process variables on RL filler performance have been produced.
2. CT/IS product has been found by B&H Canada to be insignificantly different subjectively and in the level of fines generation from control product. An economic evaluation of the CT/IS process is being made by B&H Canada for their application. They are seeking a vendor to grind their CT for further CT/IS trials prior to Manufacturing Engineering recommending a grinder for their installation.
3. Initial trials for CT application to bright and Oriental strip indicate good adhesion through cigarette making (80%) and a lower filling power (control 33.2 cc/10 gm, test 31.5 cc/10 gm, both at 12% eq. OV).
4. The first satisfactory trial of CT application to ES (after expansion) showed only 60% CT adhesion through cigarette making and a reduced filling power (control 44 ccs/10 gm; test 41 ccs/10 gm both at 14% eq. OV).

C. Plans

1. Produce coated and sized RL products from Park 500 materials for comparison with pilot plant product.
2. Complete the analysis of RL coated sheet filler properties.
3. Produce coated RL product for evaluation of product handling from hogsheads.
4. Install a dry CT distributor over the RL Foundrinier for process and product evaluation.
5. Run further trials of CT application to bright and Oriental strip and ES to establish statistically reliable data.

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II. MODIFIED RECON TOBACCO MATERIALS

A. Objective

To improve the physical characteristics of recon tobacco materials and modify its subjective character.

B. Status

1. Using a master batch of feedstock, shredded bright and burley stem products have been made by various processes [ES, IS (B&H), atmospheric and pressurized Bauer refining, and by the Rothmans shredding process]. The stem products are being evaluated for International application.
2. RL hand sheets were prepared to compare a wide range of wood pulp samples subjectively for the selection of a candidate for a Park 500 trial. Abatibi Kraft is preferred to Donitar Kraft and sulphite pulps in pilot plant sheets at 6.8% in the RL blend.
3. Burley transfer RL was preferred subjectively over this sheet with burley solubles returned after treatment by deionization and electrodialysis (27% blend).

C. Plans

1. Complete evaluation of shredded stem processes and products.
2. Obtain direction from the Flavor Development Division for further work to create RL with novel subjective characteristics.

/dbb



2022203091

CHARGE NO: 1503
PROGRAM TITLE: Modified Smoking Materials
PERIOD COVERED: February 1-28, 1983
PROJECT LEADER: G. D. Keritsis
WRITTEN BY: S. E. Wrenn

I. Salt Casing (J. Leik)¹

Objective: Determine effect of salt casings of MF filler on smoke components.

Status: It has been demonstrated that delivery of smoke components (FTC tars, NO, CO, HCN, RCHO, nicotine) can be altered by addition to the filler of monovalent cations, K^+ and Na^+ , and divalent cations, Ca^{+2} and Mg^{+2} .

Plans: A report will be issued with detailed results of this study.

II. Tobacco Extrusion (G. H. Burnett)²

Objective: Extrude a foamed, low density tobacco rod using laboratory (small scale) extruders.

Status: Trials using the Wayne plastic extruder with various screw configurations and operating conditions were not successful in producing a foamed rod.

Plans: Another small extruder will be evaluated for lab scale formulation studies.

III. CT Treatments (S. E. Wrenn, J. Leik)³

Objective: Evaluate steam pressure treated class tobacco for application onto other tobacco material.

Status: Pectin isolated from steam pressure treated class tobacco has a lower relative viscosity than pectin from the untreated tobacco. Because the pectin of the treated material has smaller chain length, it may be more soluble, increasing the "gummy" character of a slurry made from class tobacco.

Plans: Evaluate adhesion of pressure treated class tobacco by spraying onto stem material.

IV. Other Studies

- Magnesium Ammonium Phosphate system when applied to tobacco sheet or filler may enhance stiffness.⁴


- SGP (starch graph polymer) sprayed onto tobacco filler tends to decrease tobacco fall-out.⁴

- Extruded tobacco rods will be characterized as to density, pore size distribution and closed/open cell volume.⁴

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REFERENCES:

1. J. W. Leik, Notebook No. 7644, pp 84
2. G. H. Burnett, Notebook No. 7786, pp 127-128, 131-133, 136
3. S. E. Wrenn, Notebook No. 7856, pp 42
J. W. Leik, Notebook No. 7644, pp 136, 137, 142
4. N. B. Rainer, Notebook No. 7697, pp 96


S. E. Wrenn

/nb

CHARGE NUMBER: 1620
PROJECT TITLE: ELECTROPHYSIOLOGICAL STUDIES
PERIOD COVERED: February 1-28, 1983
PROJECT LEADER: F. P. Gullotta
DATE OF REPORT: March 3, 1983

I. OLFACTORY EVOKED POTENTIALS (OEPs)

OEPs have been obtained to several concentrations of methyl salicylate and to the methyl salicylate-CO₂ combination. As concentration is increased, it appears that OEP latencies decrease and that amplitudes increase. When CO₂ is added to a medium concentration of methyl salicylate, the OEP resembles that obtained to a high concentration of methyl salicylate.

The effects of stimulus duration were investigated in one subject. Methyl salicylate in pulses of 100, 200 and 300 msec. were employed. Although psychophysical judgments of odor intensity increased with increasing stimulus durations, the OEPs were little affected in latency, amplitude or waveform morphology.

II. ELECTROTRIGEMINOGRAMS (ETGs)

Recently, difficulties have been encountered in recording ETGs. The probable cause of these difficulties is crystallization of sodium chloride on the silver-silver chloride electrodes. To avoid this problem, it will be necessary to prepare fresh electrodes shortly before each experiment.

III. PARAMETRIC STUDIES: PATTERN REVERSAL EVOKED POTENTIALS (PREPs)

The Smoking Over a Four Hour Period study has been completed, and statistical analyses are in progress.

nwp

F. P. Gullotta

2022203094

PROJECT CHARGE: 1702

PROJECT TITLE: FILTRATION PHYSICS

PROJECT LEADER: R. W. Dwyer

PERIOD COVERED: February 1 - 28, 1983

DATE OF REPORT: March 8, 1983

CIGARETTE DESIGN

At Cliff Lilly's request we constructed cigarettes with special flavors. The flavors were recommended and provided by Rett Southwick. They were impregnated into 15 mm paper tubes which were inserted into tobacco rods near the filter end. The objective was to provide samples with conventional tobacco flavor on all but the last puffs. Both thymol glycoside and W.S. flavors were tested and found to provide dramatic last-puff flavor notes. The W.S. flavor in particular was not discernible until the coal reached the impregnated tube. This study is continuing. (Creamer, Dwyer)

Four samples of MUL cigarettes were prepared with paper tube inserts in the tobacco rods. Two samples contained hollow tubes of different dimensions, one contained solid tubes, and the fourth was a control. Light extinction measurements showed all samples to deliver similar amounts of TPM but their puff profiles differed significantly. These cigarettes will be smoked by in-house panelists to determine the optimum tube dimensions for early puff enhancement. The solid tube samples will be examined for flavor off-notes arising from the paper insert. (Creamer)

In cooperation with the New Products Development Project we are examining the physical properties of a variety of novel filters. These designs include paper filters, high crimp-density CA filters, and uniquely shaped CA filters. We are concentrating on the oval cigarette at this time. The exit smoke patterns of this cigarette and a control have been monitored with high-speed cinematography. There are significant differences between the smoke patterns of these samples at flow rates from 1000 to 2000 cc/min. Additionally, we are measuring their dilution and RTD behavior as functions of flow rate. The particle size distributions of the emerging smoke and the relative efficiencies of both filters are also being measured. (Fleming, Akers, Cox)

Test methods have been devised to accurately measure the role of cigarette wrapper dilution to the total cigarette dilution and RTD. A mathematical model of this contribution to smoke delivery has been derived. (Cox, Akers)

A detailed treatment of the effects of tipping paper and plug wrap porosity on filter ventilation is in progress with the Development Smoke Studies Project. A semiempirical technique was developed which allows the ventilation level of a cigarette to be predicted from the tipping paper RTD,

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the plug wrap porosity, and the filter and rod RTD's. Further samples are being prepared to extend the range of this method. (Dwyer, Cox, Akers)

A cooperative investigation of lit-cigarette ventilation and CO delivery is being performed with C. Harward and M. Parrish of the Analytical Research Division. We have characterized the effects of the coal RTD on dilution level of cigarettes on a puff-by-puff basis. A model of CO generation and diffusion through the rod wrapper has been formulated which shows excellent correlation with their CO delivery measurements. (Dwyer)

REFERENCES

C. H. Akers
K. A. Cox
R. M. Creamer
R. W. Dwyer
M. L. Fleming
L. A. Haws

RWDwyer

/ev

2022203096

PROJECT CHARGE: 1703

PROJECT TITLE: CIGARETTE MAKING TECHNOLOGY

PROJECT LEADER: A. Robinson

PERIOD COVERED: February 1 - 28, 1983

DATE OF REPORT: March 10, 1983

ADHESIVES TECHNOLOGY (A. Robinson and V. P. Henderson)

Adhesives development-formulation program

Commercially available adhesive base-emulsions formulated with varying amounts of selected compatible plasticizers were examined for their thermal and adhesive-paper interactions property behavior characteristics.

Work is continuing to determine these property characteristics in similarly prepared adhesive systems incorporated with different amounts and/or kind of boration and the degree of hydrolysis of the poly(vinyl alcohol) used as the emulsion-particles colloidal protective agent.

ADHESIVES SCREENING TEST (T. E. Majewski, T. V. Van Auken and A. Robinson)

Development of viable incoming adhesives screening tools

Data supplied by the Kimberly-Clark Corporation from their evaluation of the Werle Tack Tester as a screening tool was analyzed. The data show that some production tipping-adhesives respond differently to different sides of unconverted tipping papers, while other tipping-adhesives are virtually insensitive to the side of the tipping paper to which they are applied^(1,2).

Plans are to further evaluate the viability of the Werle Tack Tester by studying the results for a greater cross-section of adhesive-paper combinations.

Initial experiments exploring the adaptability of our mechanical testing instrument as a tack test for adhesives were performed.

Initial indications are with some modifications the instrument is adaptable.

ADHESIVES PROPERTY STUDIES (T. E. Majewski, T. V. Van Auken, A. Robinson and V. P. Henderson)

Development of adhesive properties-performance relationship data that will enable PM to specify to adhesive vendors those adhesive properties which are required to achieve optimum adhesives performance efficiency.

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Data from adhesive surface behavior studies are being analyzed with respect to adhesive emulsions tensile strength and elasticity.

Initial results suggest this kind of analysis provides insight into how efficiently adhesives with certain property characteristics can be applied during roll-application of adhesives operations.

Adhesive mixtures continue to be examined rheologically as a function of amount and/or kind of components (e.g., plasticizers, surfactants, etc.).

Studies on the testing of adhesives bond quality and strength continue on a variety of paper substrates as a function of adhesives rheology and ingredients.

REFERENCES

- 1) T. V. Van Auken, letter to Art Aronoff, Kimberly-Clark Corp., February 14, 1983.
- 2) T. V. Van Auken, Evaluation of Werle Tack Tester - trip to Kimberly-Clark (Schweitzer) in Roswell, Georgia, memo to A. Robinson, February 9, 1983.



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2022203098

PROJECT CHARGE: 1706
PROJECT TITLE: TOBACCO PHYSICS
PROJECT LEADER: D. B. Losee
PERIOD COVERED: February 1 - 28, 1983
DATE OF REPORT: March 10, 1983

TOBACCO MINIMIZATION (D. B. Losee, C. O. Tiller, P. A. Wilson)

The possibility of "selectively" cleaving bonds in tobacco carbohydrates through UV irradiation is being explored. If, for example, a percentage of glycosidic bonds could be selectively cleaved in cellulose prior to thermal decomposition, the potential exists for subjective improvement of this material. This concept has been tested by irradiating "as is" Whatman filter paper with 1200 watts of 220-180 nm light for 24 hours. The temperature did not exceed 50°C during this time period. After this irradiation the cellulose has a distinctive aroma, has yellowed (band at 370 nm) and thermally decomposes differently from control cellulose. Interestingly, if the Whatman paper is first saturated with distilled water and then irradiated under identical conditions, it breaks down differently to produce a matrix which has a distinct acetic acid aroma after irradiation. These reactions are unquestionably free radical driven and will be pursued further. A quartz reactor is being designed which will allow a greater percentage of the surface area of cellulose to be exposed to the radiation.

It has been established from Analytical testing that with the use of the appropriate porosity paper it will be possible to design a cigarette with marketplace specifications which can deliver 15 mg of tar from only 300 mg of a tobacco blend. It, of course, remains to be seen if an appropriate filler comprising 700 mg of this cigarette can be found which will not subjectively detract from the product.

As mentioned in previous monthly reports, starch (tobacco "starch") has been considered as a tobacco substitute. Equipment necessary for sheet casting starch has been obtained from Development (Mr. George Burnett) and has been installed in the lab. Several sheets of both starch and pectin have been cast to gain familiarity with the equipment and the techniques required.

The thermal analyses of the white puffed part of popped popcorn and cornstarch have been completed. The EGA/MS data for popcorn are being plotted and interpreted.

Another tobacco substitute that has been investigated is kudzu. The thermal analysis of kudzu under pyrolysis and combustion was completed and compared with bright tobacco. If green kudzu is compared to cured (dried) kudzu, a new exothermic decomposition pathway appears at approximately 420°C. Additionally, kudzu does not display a sugar decomposition peak as

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does bright tobacco. A reduction (or absence) in pectin and hemicellulose decomposition when compared with bright tobacco is also evident.

MECHANISMS OF WET EXPANSION (B. E. Wyamack, K. R. Squire)

EGA/MS runs were completed in the Entrained Flow Reactor on uncased bright filler and water extracted filler. The runs were performed at 650°F in steam. Concentrations were measured at a residence time of ~ .45 sec. Consistently higher levels of CH₃OH and CO₂ were evolved from the unwashed filler. These results agree with previous tube furnace comparisons of the same tobaccos after 3" tower WET processing.

/ev

D.E. Squire

2022203100

CHARGE NUMBER: Project 1708
PROJECT TITLE: Physical and Chemical Properties of Tobacco
PERIOD COVERED: February 1-28, 1983
PROJECT LEADER: B. C. LaRoy
DATE OF REPORT: March 3, 1983

PARTITIONING OF WATER IN STEM AND LAMINA^{1,2} (J. Crump, L. Trentham)

A study of the kinetics of water sorption by "factory stem", "factory lamina", whole leaf, "test stem" and "test lamina" is in progress. Factory stem and lamina designate material separated by the Richmond Stemmary. Test stem and lamina designate material obtained from the Stemmary as whole leaf, reordered as whole leaf then hand separated for analysis. Initial results on Burley grade B57 give adsorption relaxation times for each sample as follows:

Factory Stem	4.9 hrs
Factory Lamina	4.0 hrs
Whole Leaf	6.7 hrs
Test Stems	9.9 hrs
Test Lamina	6.0 hrs

Sorption rates for factory separated stem and lamina are essentially the same and are faster than for whole leaf. The slower rate of sorption for stem compared with lamina when they are part of a whole leaf could prove useful during Stemmary threshing. If the same is true for desorption, it may be possible (as suggested by J. Whidby) to overwet leaf and then rapidly dry it back to the normal OV (approximately 18%) just prior to threshing, leaving stems more moist than the lamina, and thus more flexible for threshing. Desorption studies are in progress.

TRACKING METHODS FOR TOBACCO ACCOUNTABILITY^{1,3,4} (M. Wood, J. Crump, H. Hartung)

For tobacco accounting purposes, it is desirable to track the mass flow of tobacco independently of water and casings. Two current methods for tracking tobacco "dry weight" are OV and GC water. The former method is substrate dependent, while the latter is more time consuming and has more measurement-to-measurement variability. It has been suggested that tracking an "inorganic" component (Ca, Mg or K) might provide a better means of accounting for tobacco loss or gain than either moisture measurement method. Thus, a cooperative program with QA and the Analytical Division was initiated for quantitative comparison of these approaches. The program includes comparisons of OV, GC water and inorganic analyses on dried tobacco to which measured amounts of water and casings have been added. Initial experiments are in progress.

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FILLER LENGTH EFFECTS ON BULK MODULUS⁵ (B. LaRoy, L. Trentham)

Following extensive repairs to the Instron machine, previous data were rechecked for accuracy. As before, DBC bright filler compressed to 0.16 g/cc could accept the admixture of 20% by weight of ground bright filler "dust" with no alteration in the pressure required for compression. For the same filler compressed to 0.2 and 0.25 g/cc, the addition of 20% dust increased pressure by approximately 20% and 25%, respectively. Work in this area is continuing with current emphasis on eliminating instrumental effects from data at densities above 0.3 g/cc.

TOBACCO BREAKAGE MECHANISMS⁶ (T. W. Haas)

Initial review of the literature has identified only one friability test for tobacco. This consists of simply chopping the tobacco shreds in the Waring Blender. However, the Kramer shear press which is used to measure the "texture" of vegetables may be of interest since it operates in a testing machine which allows quantitative measurements of forces and energies. The data obtained might compliment results currently being developed by Project 0307 with the Hobart food cutter.

Preliminary experiments with rectangular samples cut from cured leaf and tested in tension in the MTS machine indicate that edge effects may be a problem since all failures initiated at the side of the specimen midway between the grips. However, additional testing will be required before any definite conclusions can be reached.

VIBRATION TESTING⁷ (D. Full)

Initial investigations are in progress to examine the acoustic emissions of cigarettes subjected to sinusoidal vibration. Preliminary results suggest that such emissions are indicative of rod defects and that defect location may be determined acoustically.

RL STIFFNESS⁸ (L. Trentham)

Stress-relaxation measurements of RL samples have been completed. Measurements of RL samples cut with the grain of the sheet and transverse to the grain of the sheet have been made. Similar measurements of RL sheet coated with class tobacco, and cut with similar orientation to the grain of the sheet, have been completed. Analysis of the data is under way.

COMPUTER MODELING⁴ (H. Hartung)

Programs for training laboratory personnel in the use of APL Cigarette Modeling capabilities were initiated jointly with the Computer Applications Division. On-line, interactive tutorials proved very successful in preliminary trials, and they turned out to be quite

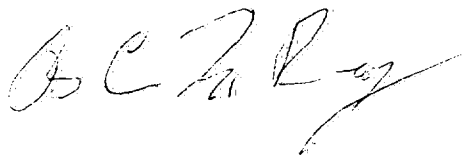
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easy to implement. We plan to pursue this approach because it offers users the opportunity to apply cigarette modeling results in their work without necessarily investing large blocks of time and effort in computer training.

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1. J. C. Crump, Notebook #7220, pp. 178-82.
2. L. M. Trentham, Notebook #7885, pp. 2-8.
3. M. J. Wood, Notebook #7653, pp. 36-37.
4. H. A. Hartung, Notebook #7805, pp. 25-30.
5. B. C. LaRoy, Notebook #7838, pp. 86-90.
6. T. W. Haas, Notebook #7676, pp. 70-72.
7. D. A. Full, Notebook #7199, p. 198.
8. L. M. Trentham, Notebook #7885, pp. 12-26.

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2022203103

CHARGE NUMBER: Project 1720
PROJECT TITLE: Microstructure Research
PERIOD COVERED: February 1-28, 1983
PROJECT LEADER: L. R. McCray
DATE OF REPORT: February 28, 1983

Air Cured Bright Tobacco

Light microscopy and scanning electron microscopy studies are in progress, and some preliminary results have been obtained:

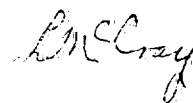
- a. Cross-sectional thickness measurements on mid-stalk position samples of ripe, end of flue curing and end of air curing indicated a decrease in thickness. Air cured samples were approximately half as thick as flue cured samples.
- b. Density of trichomes and stomata per unit area was studied to determine the degree of lateral shrinkage of the leaf as a result of curing. Minimal changes were observed between ripe, flue and air cured samples, which indicate little, if any, lateral shrinkage during curing.

The prevalence of mold on/in air cured tobacco as compared to flue cured tobacco is being investigated.

Image Analysis

The "IBAS" image analysis system, distributed by Zeiss, Inc., is being evaluated.

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2022203104

CHARGE NUMBER: Project 1730
PROJECT TITLE: Plant Tissue Culture
PERIOD COVERED: February 1-28, 1983
PROJECT LEADER: L. Weissbecker
DATE OF REPORT: February 28, 1983

I. TOBACCO PLANT PROPAGATION

Havana 38 callus, started from a greenhouse-grown plant, has been made to differentiate and form shoots. These shoots are beginning to form roots with high auxin stimulation. The conditions required to form plantlets from callus tissue are somewhat more rigorous than those used with cotyledons.

Plans: Transfer plantlets to pots in the greenhouse and allow to reach maturity.

II. GROWTH OF TOBACCO CELLS

Undifferentiated cells have continued to grow in suspension culture. The cells have a typical "S" shaped growth curve with a lag phase, log phase and plateau. The lag phase was shortened by repeated subculturing during log phase. Cell wet weight doubling time in log phase was reduced to 47 hours.

III. TOBACCO BY-PRODUCTS

Investigations into secondary products from plant tissue culture have begun. Noel Einolf¹ analyzed several samples for sucrose esters. Undifferentiated callus tissue, grown in the dark, had no detectable sucrose esters. Callus, grown under light, turned green, indicating chlorophyll production, began to form shoots and produced some sucrose esters. Shoots of Havana 38 tobacco initiated from cotyledons and meristem also contained sucrose esters. The samples contained about one-tenth the amount of sucrose esters found in leaves from greenhouse-grown Havana 38.

Plans: Attempts will be made to try to increase sucrose ester production.

In vitro initiated Havana 38 shoots have trichomes on their leaves and stems. Samples of these plantlets were run on TLC plates by Rett Southwick² for cis abienol. Although some material migrated on the plate, its Rf did not correspond to cis

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abienol or duvatrienediol. Leaves of greenhouse-grown Havana 38 plants did not contain cis abienol either.

Plans: Roger Bass will get seeds of tobacco plants, reported in the literature, to contain cis abienol. Cells of these plants will be grown and analyzed for cis abienol.

IV. REFERENCE

1. Noel Einolf, Notebook #7853.
2. Rett Southwick, Personal Communication.

L. V. Southwick

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2022203106

PROJECT NUMBER: 1801

PROJECT TITLE : Expanded Tobacco Development

PERIOD COVERED: February 1-28, 1983

PROJECT LEADER: F. V. Utsch

I. REORDERING OF DIET (P. E. Aument)

A. Objective

Develop a reordering system using water mist, humid air or steam application that could be incorporated into the production facilities to improve expanded filler shred length without excessive filling power loss.

B. Status

1. Vibrating Conveyor Spray Reordering

Off-line tests on the vibrating conveyor unit have been completed and the data is being summarized. The results show that the use of the spray reordering system gave filling power results comparable to those of the cylinder without the excessive breakage of the cylinder. Buildup due to overspray accumulation remains a problem.

2. Vibranetics Unit

Modifications to the Vibranetics for steam reordering have been completed and testing is underway. Preliminary results indicate that the use of low temperature steam in the Vibranetics fluidized bed unit rapidly reorders DIET without breakage, but with equal or greater filling power loss than the reordering cylinder.

C. Plans

Vibranetics fluidized bed steam reordering tests will continue. Humid air reordering tests are also planned. Vibrating conveyor spray reordering tests are planned in-line with the Phase III/8" tower system.

II. HUMIDIZONE SYSTEM FOR DIET REORDERING (D. C. Haller)

A. Objective

Evaluate the Wolverine Corporation Humidizone reordering concept for DIET.

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B. Status

The quote from Wolverine Corporation for a 200 pound per hour pilot unit was over \$100,000. The decision was made, therefore, to evaluate the temperature and humidity characteristics of the system on a prototype batch unit.

Hook-up of a modified blow box with the 1000 and 300 cfm Aminco-air units is almost complete. The Aminco-air units will provide humid air for bottom to top flow reordering on a perforated plate tray (8% open area).

The climatelab cabinet is being modified for fundamental humidity/reordering studies using a steam/water mixing valve to provide controlled changes to the humidity profile. Preliminary tests with the unit before modification showed the capability of reordering to 11% OV in 5 minutes at 178°F and 80% RH. (Target conditions for prototype are 180°F, 80% RH @ 5 minutes.)

C. Plans

1. Begin test grid with blow box/Aminco immediately.
2. Climatelab being modified; Engineering Services to provide estimate for programmable control capability.
3. Continue to investigate vibrating conveyor to handle 8" tower exit with 5 minute residence time.
4. Hook-up Aminco unit to Vibranetics conveyor.

III. EXPANDED FILLER BREAKAGE (R. Z. Burde, P. E. Aument)

A. Objective

To determine the nature and effect of filler breakage as it is processed through the steps of the DIET process and to determine the best methods to improve the filler shred length.

B. Status

Based on the results of the first part of the breakage study, the testing was extended to define the nature of the improved sieve analysis due to expansion of shorts and mediums in the feed tobacco. DBC bright filler has been processed through the maker to simulate the shred breakage in the clumpbreaking phase of the DIET process. Separation into the various sieve fractions has begun.

C. Plans

The various sieve fractions will be expanded via the DIET process. Cigarettes will be made utilizing the longs from each of the expanded sieve fractions. Cigarette firmness and coal strength characteristics will be related to the origin of the long fibers.

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IV. NONTOWER EXPANSION - VIBRANETICS CONVEYOR (D. C. Haller)

A. Objective

Determine feasibility of expanding tobacco on a perforated-plate vibrating conveyor.

B. Status

Results of three test runs show filling power gains comparable to those of tower processing. The standard product curve for the Vibranetics falls close to the tower processing curve with a 0.25% equilibrated OV shift to the left. There is little or no measurable breakage of DIET in the conveyor. Some clumps of impregnated filler still existed in the product.

C. Plans

Further testing to determine optimum conditions of gas velocity, feedrate and residence time are planned after the steam reordering tests are concluded.

V. "POP-GUN" PUFFING OF TOBACCO (G. W. Stamper)

A. Objective

Evaluate direct injection of gas or liquid impregnated tobacco into an expansion tower from a pressurized chamber mounted directly on the tower in order to capture the shred expansion which occurs on release of the chamber pressure and/or due to heating.

B. Status

The jacketed pop-gun was installed and initial testing both with and without cooling on the jacket revealed excessive breakage at 100 psi and 200 psi. Close inspection showed a 1/4" shoulder in the new pop-gun, due to a difference in pipe schedule from that in the old unit.

C. Plans

Reinstall the old unit to verify that product breakage is not excessive with that system; change the pipe thickness on the jacketed unit to eliminate the internal shoulder.

VI. IMPREGNATION WITH CONTROLLED CO₂ UPTAKE (G. W. STAMPER)

A. Objective

Apply existing impregnation technology to achieve controlled CO₂ retention and eliminate clumping.

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B. Status

The sparge tubes have been received. New baskets are being fabricated and pipe fittings are on order. Initial atmospheric testing with CO₂ gas in the old baskets indicates that a good radius of penetration (6") and uniformity ($\pm 3^{\circ}\text{F}$) can be achieved.

Fill and vent rates for CO₂ gas from 200 psi to 800 psi were measured using the existing piping arrangement in the Phase II. A vent time of up to 38 seconds was recorded. This reveals the need to modify the Phase II piping for faster fill and vent rates.

C. Plans

1. Install the sparge tube and begin testing for radius of penetration.
2. Modify Phase II piping for faster vent.
3. Perform presnowing tests in the Phase II unit at 500 psig.

VII. IMPROVEMENT IN STEMMERY OPERATION (R. Z. Burde)

A. Objective

Assist in the development of methods to prevent folding of strip on drying and attempt to increase the "large" sieve fraction.

B. Status

Microwave drying eliminated the necessity of overdrying burley strip to achieve uniformity. In spite of that, there was no improvement in the large fraction retained on the top screen. Apparently, overdrying is not the key factor causing the folding of strip. It was postulated that perhaps the rapid rate of microwave drying caused additional stress in the leaf which led to "folding." To test this hypothesis, two slow drying rates (10 and 18 minutes) were tested in a laboratory oven. The results indicate that the same degree of "folding" still took place. Apparently the rate of drying is also not the determinant factor. Microscopic evaluation of strip dried by various techniques confirmed the visual observations.

It was observed that the strip held under mild pressure during drying did not "fold." We attempted to induce that phenomenon by drying strip under pressure on a Yankee dryer, and in a lab oven while pressed between two screens. In both cases a flat strip which did not "fold" after prolonged storage was obtained. The strip was cut using the guillotine hand cutter. The results of the 3 tests indicate that in hand operation, the content of longs does not increase by drying strip under pressure. The retention of strip on the top screen appears somewhat larger, but this results probably cannot be translated into an increased level of long fibers after cutting. It is not known if prizing of flat strip would play an important role in its behavior on cutting.

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C. Plans

Issue two memoranda summarizing the study on drying of strip by microwaves and on the drying of "pressed" strip. It is recommended that a study be done by the Stemmary Task Force on the structural differences between air dried control strip and the P&S samples.

VIII. WET PROCESS WITH A "KICK" (R. Z. Burde)

A. Objective

Establish if gaseous CO₂ addition to water prior to moisturizing under pressure can improve the filling power of the WET product.

B. Status

An idea of Jerry Osmalov of spraying CO₂ saturated water onto tobacco has been tested. To increase the CO₂ content (supersaturation) and to minimize the CO₂ loss in spraying, the technique has been modified to include spraying into a container under a CO₂ pressure of 20 lbs.

Two series of runs were completed. In the first series the addition of water was non-uniform and led to widely different results. The second series contained 15 samples with target moistures of 14 and 20% OV. The technique is greatly improved, but it is still not possible to establish if a true upward directional move exists in CV's. At best, several CV units might be obtainable by this technique.

C. Plans

Repeat the test using a larger number of samples. Search for technique which would allow for mixing of the sample under pressure, while the addition of water takes place. Laboratory pressure rotary cylinder would be a perfect equipment for the above mode of experimentation.

IX. ALTERNATE IMPREGNATIONS (R. Z. Burde, B. A. Vaughan)

A. Objective

Develop new concepts of impregnation and formulate commercially feasible alternatives to the present DIET process.

B. Status

It has been shown that fluctuation of pressure around the triple point did not increase the gaseous penetration and deposition. The same results were obtained when the experiments were conducted in CO₂ liquid and the liquid was allowed to drain prior to pressure fluctuation. It appeared that the pressure fluctuation actually stripped the impregnants from tobacco fibers. We started evaluating if such a concept could lead to decreasing the clump hardness on the Phase III unit. To obtain clumps with a hardness comparable to that at the MC, 130 lb batches are being studied.

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No expansion was obtained when the filler and liquid CO₂ temperature was kept at approximately "200 psig equivalent" and yet the pressure was boosted by gas to 600 psig. Such product, which was basically exposed to "artificially" low temperature and high pressure retained high levels of CO₂ but the impregnant was retained on the fiber surface. It appears that CO₂ film which formed on the surface of the filler "crackled" (i.e. burst) when exposed to slightly elevated temperatures.

Conversely, the use of "artificially" higher temperatures at lower than conventional pressures was also tested. Using filler preheated by dielectrics, we observed that a suitable impregnation might be possible at somewhat lower pressures (approximately 100 to 200 psig decrease). Unfortunately it appears that the allowable decrease in pressure is too small to permit effective low pressure impregnation at this time. Use of other variables will be sought which in conjunction with the use of preheated tobacco could lead to a continuous low pressure commercial process.

C. Plans

Complete preliminary study of triple point fluctuations as a means of decreasing the DIET clump hardness. Release memo summarizing the progress of work on alternate impregnations to date.

Design program for detailed study of the "hydrate process" and the variables which would allow for decreasing the processing time. Study the effect of autoclaved (precooked) filler on the progress of gaseous and liquid CO₂ low pressure impregnation.

X. SUPERCRITICAL PROCESSING (R. Z. Burde)

A. Objective

Reproduce the patent of Reemtsma for the N₂ and Argon impregnation at approximately 10,000 psig. Determine if the technology has any possible application for PM.

B. Status

We will not be allowed to use the large equipment at USDA because of their long term work on highly flavorful substances. However, a suitable pressure, 2 liter unit was located. A new director was just appointed to the USDA station and we must wait for his approval, which should be forthcoming in the next two weeks.

A memorandum of understanding which we are required to sign was requested for review by the attorneys.

We have designed a baffle which converts the conventional laboratory oven into a "transportable tower."

C. Plans

Study the conditions under which the "transportable tower" can best simulate our expansion tower. Review the memorandum; schedule the trip.

J.V. Hark

2022203112

PROJECT NUMBER: 1804

PROJECT TITLE : Thermal Treatment Processes

PERIOD COVERED: February 1-28, 1983

PROJECT LEADER: R. G. Uhl

OBJECTIVE

Develop thermal treatment technology to improve the filling power, piece size and modify the subjective response of tobacco.

STATUS

1. Trials were continued to characterize and determine the limitations of the 4" horizontal tower. Five quench nozzles were evaluated, with nozzle size and process gas temperature being varied. Wet spots in the product were only encountered with a spray nozzle installed at the separator transition piece. Results confirmed preliminary indications. High water spray rates stop thermal treatment in the reordering zone and limit product CV, indicating the need for more tower length. Product CV's of 50-55 are obtained with in-situ reordering to 10-11% OV, while normal CV (75) is achieved with product reordered to only 5-6% OV.
2. Overall sieve results show an advantage with the horizontal tower. Average tower exit values using the "new" %L/%L+M (>6 mesh/total >12 mesh) are 21/60 for the horizontal tower versus 17/55 for the pilot 8" tower and 12/52 for MC/DET this quarter.
3. Four spray nozzles were installed on the 8" tower (one at each elbow; two in the riser) to evaluate in-situ reordering on a conventional expansion system. CV results approximated horizontal tower results, showing the same dependence on spray rate (49 CV at 11% tower exit OV; 76 CV at 5% tower exit OV). Tower exit longs (12-14%) were lower than conventional product. Nozzle size at the first (lower) elbow had to be limited to prevent passage of ice balls and wet clumps through the tower and drastic loss of product CV, indicating that little expansion had taken place up to this point in the system.
4. Small (25 lb) batches impregnated in Phase III were soft enough to be processed without the clumpbreaker. However, this material showed little improvement in longs fraction at the 8" tower inlet valve, and yielded only a small improvement in the tower exit piece size (%L/%L+M = 18/59).
5. CAR unit treatment of 20% OV uncased bright strip showed several disadvantages versus the treatment of cut tobacco at the same conditions. The strip product had gross non-uniformities in reducing sugar and alkaloid content. This is attributed to reduced internal air circulation with strip. Also, CV gain was only half the cut tobacco value, indicating that strip would require significantly longer treatment times to effect a given subjective change.

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6. Overall average sieve results for CAR treated cut bright show that long fraction loss during CAR treatment is comparable to the loss encountered in tower expansion systems.
7. CAR treated bright tobaccos taken to the same CV endpoint by different time/temperature profiles are currently being subjectively screened in 100% and blended cigarette models.
8. Marlboro cut filler (12% OV) was CAR treated at the maximum heating rate (5 F°/min). CV increased 12 units in 15 minutes of heat treatment, with no additional increase at longer times. Shorter treatment times will be investigated prior to subjective testing.
9. The initial microwave oven simulation of the roaster drying process was completed. Marlboro cut rag showed a small advantage (up to 1 CV unit) attributable to superwetting to 30 and 40% OV. Maximum CV results were obtained with constant moisture bulking at 200°F. Longer bulking times will be examined to determine if the maximum effect has been attained.

PLANS

1. Prepare design proposal and initiate installation of a longer horizontal expansion tower.
2. Evaluate the effect of cut width on expanded product piece size for conventional and horizontal towers.
3. Evaluate gaseous impregnation to determine the effect on the piece size of impregnated feedstock and expanded product.
4. Replace the center mounted spray nozzles on the horizontal tower with nozzles mounted flush with the pipe wall and evaluate the effect on product breakage.
5. Evaluate the effect of casings on CAR product CV's and have cigarettes made for subjective comparison.
6. Determine the feasibility of blending CAR treated cut bright into the final cut blend.
7. Evaluate short CAR treatment times for MF cut filler.
8. Microwave roaster simulation:
 - a. Evaluate longer superwet bulking times.
 - b. Define maximum CV procedure.
 - c. Initiate testing of factory MF cut rag.
 - d. Initiate testing of individual blend components.

/dbb

R. L. J. M.

2022203114

CHARGE NUMBER: Project 1901
PROJECT TITLE: Biochemical Modification of Tobacco
PERIOD COVERED: February 1-28, 1983
WRITTEN BY: D. M. Teng
DATE OF REPORT: March 7, 1983

AIR-CURED TOBACCO

The 1982 crops of air-cured and flue-cured tobaccos from Southern Piedmont Center were stemmed at the Stemmary. They were submitted for chemical analysis, expansion and cigarette making.

Bright tobacco samples from different stages of flue curing and air curing were analyzed. In flue curing, the α -amino-N increased during the yellowing stage, followed by a slight decrease until the end of curing. No soluble-ammonia-N was detected. Increase in total reducing sugars was detected from the yellowing stage. In air curing, the α -amino-N was highest at the end of the second week, followed by a slight decrease at the end of curing. Increase in soluble-ammonia-N was noticed at the end of the fourth week. This clearly indicates that the concentration of soluble-ammonia-N is influenced by the curing method used. A possible source of soluble-ammonia-N could be from the degradation of amino acids. Air curing/flue curing of burley tobacco should reveal the influence of tobacco type and curing method on soluble-ammonia-N concentration in cured tobacco. In air curing, the total reducing sugars peaked at the end of the first week, followed by a steady decrease until the end of the curing.

OTHER STUDIES

1. Costa Rican Sun-Cured

Four hogsheads of Costa Rican sun-cured tobacco were received. Samples were submitted for chemical analysis and cigarette making.

2. Other Tobacco Materials

Several tobacco samples were received from the Leaf Department. They were submitted for chemical analysis and cigarette making.

D. M. Teng

/lvb

2022203115

CHARGE NUMBER: Project 1902
PROJECT TITLE: Microbial Technology
PERIOD COVERED: February 1-28, 1983
PROJECT LEADER: V. S. Malik *VS Malik*
DATE OF REPORT: February 3, 1983

BIOTRANSFORMATION OF IONONES (J. Lyle, D. Christmann)

Selected isolates of molds, bacteria, yeasts, fungi and streptomycetes were grown in various media. Beta and alpha ionones were added to the growing cultures after the second day of inoculation. Broths of growing organisms were extracted with methylene chloride at regular intervals for a period of three weeks and methylene chloride phase examined by liquid gas chromatography. This experiment has been completed on about two dozen isolates to obtain kinetics of products of biotransformation of ionones. These selected cultures will be further examined for their ability to modify ionones and will be sent to American Type Culture Collection for taxonomic placement.

BIOTRANSFORMATION OF MENTHOL (V. Malik)

Menthol was added to growing cultures and the broths examined for the biotransformation product of menthol. Several organisms have been selected that modify menthol into novel derivatives. These microbially modified menthols may be of value in enhancing the flavor properties of smoke.

VIABILITY OF CULTURES (J. Lyle, D. Christmann)

The valuable cultures have been restreaked to check their purity and viability. The work on storage and maintenance of cultures was performed as usual.

/llvb

2022203116

CHARGE NUMBER: Project 1904
PROJECT TITLE: Bioengineering Studies (Process)
PERIOD COVERED: February 1-28, 1983
PROJECT LEADER: J. L. Banyasz
DATE OF REPORT: February 28, 1983

PILOT PLANT

During February, the design of the NOD pilot plant retrofit was finalized. Construction of the continuous flow sterilizer has been completed, and the piping of the other equipment has begun. The new deadline for completion of construction is March 14.

Personnel selection for the pilot plant is now under way. Training of operators is expected to start on March 14 and to last for two weeks. Work is now under way to complete the training package.

Work is also under way for the layout, equipping and staffing of a pilot plant laboratory to provide analytical and microbiological support to the pilot plant.

/lvb

J. L. Banyasz

2022203117

CHARGE NUMBER: Project 1904
PROJECT TITLE: Bioengineering Studies (Laboratory)
PERIOD COVERED: February 1-28, 1983
PROJECT LEADER: I. L. Uydess
DATE OF REPORT: March 4, 1983

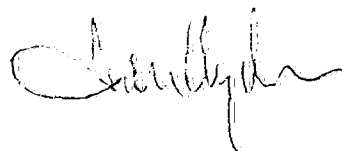
R. Dunn, P. Oglesby and other Project 1904 personnel are continuing investigations to further elucidate NOD process parameters as guidelines for the design and engineering of the Park 500 NOD facility.

P. Kusel is continuing his examination of the physiological basis for dissimilatory denitrification as exhibited by the mixed culture in both SEL and SEL-equivalent (defined) media.

D. Chadick and S. Tenhet collaborated with E. Taylor and R. McCuen in demonstrating the presence of ATCC-1-equivalent organisms in the mixed culture (by agglutination) using specific antibody to ATCC-1 made by PM personnel.

Pilot plant renovations are on schedule and should be completed by late March. The initial testing of the NOD process should begin in April.

/lvb



2022203118

CHARGE NUMBER: 1990
PROJECT TITLE: Blend Development
PROJECT LEADER: C. Moogalian
PERIOD COVERED: February, 1983

I. SPECIAL TOBACCOS

Objective:

To determine the character and possible utility of tobaccos that are atypical of current usage.

Status/Plans:

POLs 2078 and 4278 have been made and will be shipped by mid-March. POL 2078 is a 5 mg menthol containing Virginia Sun-Cured (VSC) and will be tested against the 9 mg Salem Lights. POL 4278 is a 5 mg nonmenthol with VSC and will be tested against the 9 mg Winston Lights.

The recent purchase of VSC tobaccos is grouped into four grades - SCBL, SCTL, SCX, and SCN. Subjective evaluations indicate all but the SCX grade to possess desirable Dark Air-Cured character. Chemical blend data is incomplete. DIET expansion of these grades is scheduled for early March. Blending studies will be done with both expanded and nonexpanded tobacco.

Subjective and chemical evaluations will be done in March with the six Green River grades in stock.

II. PROJECT TOMORROW

Objective:

To develop a prototype preferred 60:40 over Marlboro by Marlboro smokers.

Status/Plans:

POL 3204 results showed a significant preference for the Y83-1 Marlboro control over the experimental among Marlboro smokers. Previously, in POL 3183, a similar experimental (contained Y79-3 by-products instead of Y83-1 by-products) was significantly preferred over the Y79-3 Marlboro by Marlboro smokers, who cited smoothness, less harshness, and better aroma in the experimental. In the current POL 3204, no significant discriminations were made by the Marlboro smokers.

POL 3204 - Preference

	<u>Control</u>	<u>Test</u>	<u>p</u>
Marlboro Smokers	57.6	42.4	.0588
Winston Smokers	51.0	49.0	.8865
Other FFX Smokers	44.7	55.3	.3703

In POL 3205, the experimental differed from the POL 3204 experimental in that the 3205 contained more thin tobaccos. The results show the control significantly preferred by Marlboro smokers, and no difference among Winston and other FFX smokers. No significant discriminations were made by either Marlboro or Winston smokers.

POLs 3232 and 3233 have been made and are awaiting analytical data. Also, it is planned to retest POL 3204 using only RL-TC (instead of the combined RLs) in both the control and experimental.

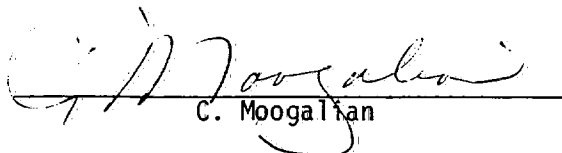
III. OTHER STUDIES

A. New Project Blends

Three blends have been designed and submitted for Project Bingo. Initial blends for Project 56 are awaiting analytical data.

B. Marlboro Study

Preliminary results from the incomplete block testing of Marlboros Y79-3, Y84-1, and Y84-1 without BL indicate no differences among the three cigarettes.


C. Moogan

CM/lad

2022203120

CHARGE NUMBER: 2100
PROJECT TITLE: New Products
PROJECT LEADER: W. G. Houck, Jr.
PERIOD COVERED: February, 1983

I. NEW PRODUCTS

A. Raffles/Players [Project Bingo] (L. Brown)

Objective:

To develop a freestanding 85/100 mm menthol product in the 12-13 mg and 14-15 mg tar range, respectively.

Status:

Test market production initiated. Possible taste differences contributed by the box pack are under investigation. HTI testing the Marlboro with B&H aftercut vs. special menthol flavor and POL testing of B&H aftercut vs. tobacco flavor in progress. HTI pack age and pack type (Westvaco vs. Tako) tests requested. POL testing requested for identified vs. unidentified packaging and cigarettes. In-house filter flavor, blend modifications, plasticizer type, and RTD studies are in progress.

Plans:

Complete HTI/POL testing	April/May, 1983
Complete flavor investigation of box	Continuous

B. Project Othello [Rothmans/P.M. Lights] (L. Brown)

Objective:

To develop a low-delivery product (11 mg 85/13 mg 100) with strong appeal to full-flavor smokers.

Status:

Completing in-house subjective evaluations

Plans:

Complete in-house subjective evaluations	March, 1983
Initiate national testing	April, 1983

C. Project DATA (R. Newsome)

Objective:

To develop an adjustable tar cigarette product.

Status:

Made initial models with higher tar range. Made models with new graphics. Planning additional focus group testing. Modified design to reduce air leakage.

Plans:

Install development module (Max S, Mark 9, laser and tray filler)	March/April, 1983
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2022203121

February, 1983

Refine designs

Continuous

Outside consumer testing

April/May, 1983

D. Project Zenith [P.M. Ovals]Objective:

To develop a free-standing filtered oval product (12 mg 85/
14 mg 100).

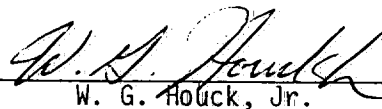
Status:

Completed initial in-house discriminative and pack hand-out
testing.

Plans:

Test market

2nd Qtr., 1983


W. G. Houck, Jr.

WGH/lad

2022203122

CHARGE NUMBER: 2105
PROJECT TITLE: Filter Development
PROJECT LEADER: Walt Nichols
PERIOD COVERED: February, 1983

I. TOBACCO EXTRUSION

Objective:

Develop an extruded foam tobacco product.

Status:

Processing parameters effecting rod density were identified. Reducing die orifice size and increasing extrudate velocity are the most significant parameters. Rod density was reduced to ≤ 0.25 g/cc, equivalent to a conventional cigarette.

Several formulations having different binder levels were extruded to determine the relationship between cell wall strength and rod pressure drop. Currently, typical rod RTD is 6 - 9 inches of H₂O (total encapsulation). A target RTD of 2 - 2.5 inches of H₂O is achievable by permitting bypass between the wrapper and rod. A less conspicuous method involving extruding a small hole down the center of the rod was tested. Unfortunately the temperature of the smoke stream was sufficient to degrade the filter. Additional testing will be done to diffuse the heat over a large surface area by using a multiplicity of holes.

Several samples were produced for subjective screening. Formulations having higher burley content are preferred for eliminating off-taste.

Extruded sections of foamed tobacco rod were evaluated as filter elements. A 27 mm, 3.75 inches of H₂O RTD section yielded a 38% efficiency. While this is lower than a cellulose acetate filter of equal RTD, it may have applications.

Plans:

Paperwrap a 0.25 g/cc tobacco rod at 16% OV	March, 1983
Produce a subjectively acceptable product	Continuous

II. FILTER DEVELOPMENT

A. Bridon Polypropylene Fibers

Objective:

Determine the process requirements for producing a subjectively acceptable Bridon polypropylene filter.

Status:

Several adhesives were evaluated for their fiber bonding capability. Filter firmness values were in all cases insufficient. This result appears to be attributable to overbloomng the tow. Complete decrimping of the fiber seems to require more force using the Hauni AF-1. The Hauni AF-2 used by Bridon apparently can bloom the tow with less fiber breakage. Modifications to the AF-1 will be made to make it replicate the action of an AF-2.

2022203123

February, 1983

Plans:

Complete adhesive evaluations

April, 1983

B. Plasticizer Applicator

Objective:

Evaluate a two-sided plasticizer applicator for filter making.

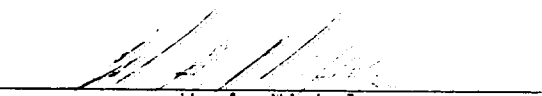
Status:

Working in collaboration with Engineering U.S.A., a two-sided plasticizer applicator has been installed on a Hauni AF-1. Evaluation will begin shortly to determine if a more uniform plasticizer application can be achieved with this method.

Plans:

Complete evaluation and transfer to Manufacturing

May, 1983



W. A. Nichols

WAN/lad

2022203124

PROJECT CHARGE: 2106

PROJECT TITLE: APPLIED TECHNOLOGY

PROJECT LEADER: P. Martin

PERIOD COVERED: February 1 - 28, 1983

DATE OF REPORT: March 9, 1983

FLAVOR MODIFICATION

Experiments were completed in which the monoammonium salt of aspartic acid was applied to bright stems and subsequently cylinder heat treated. The sample with salt addition was not preferred to a bright stem control, but the heat treated sample was judged preferable to the control. It was characterized as being smoother and having more tobacco character.

Two collaborative projects were initiated with Product Development to test the ammoniated bright tobacco.

FLAVOR IDENTIFICATION

The water extract of burley tobacco has been run through a preparative scale Shodex column. The resultant fractions will be concentrated and tested on handmade cigarettes.

Ammoniated bright tobacco cigarettes were smoked through a long charcoal trap and were judged very mild. Tests are now being run to determine the removal efficiency of the column for gas phase and particulate components.

PACK SEALING

Sets of packs have been sealed to pack pressures in excess of 100. These have been submitted for storage testing under jungle and desert conditions. This test was not done before since the IMPS device could only measure up to 100.

Measurements are being made regularly on a series of packs to determine the inherent variability of the measuring technique.

The production IMPS units will not be available until mid-March because of minor machining problems.

OTHER PROJECTS

A method has been developed for measuring lip position on cigarettes. O-Phthalaldehyde is used to derivitize the primary and secondary amines deposited on the filter by the lips. The cigarette is then placed under UV light and the derivitized material fluoresces to show the lip position.

/ev

P. Martin
ev

2022203125

CHARGE NUMBER: 2304
PROJECT TITLE: FLAVOR CREATION
PERIOD COVERED: FEBRUARY, 1983
DATE OF REPORT: MARCH 1, 1983
WRITTEN BY: K. PARRISH, J. ETHERIDGE, A. VULOVIC

I. Ammonia In Cigarettes

OBJECTIVE:

To determine the optimum level of ammonia in cigarettes.

CURRENT STATUS:

Cigarette models constructed using DAP as the ammonia source were analytically and subjectively evaluated. The DAP was applied to RL-TC at levels from 0 to 8%. The subjective findings were confusing at best. Additional models using BL, RL-TC and specially treated RL-TC have been requested.

PLANS & TIMETABLE:

New models have been requested in semi-works. Consumer research testing will be requested after evaluation of these models - March, 1983

II. Sweet Flavor

OBJECTIVE:

To develop a coumarin type flavor.

CURRENT STATUS:

Several models have been constructed by semi-works and internally screened for proper flavor level. Chosen models were submitted to C.E.S. for subjective evaluation.

PLANS & TIMETABLE:

Based upon the results from C.E.S., mail-out consumer tests will be initiated in March, 1983.

III. Burley Type Flavor

OBJECTIVE:

To develop a flavor that fortifies the subjective contribution of burley in a blended cigarette.

2022203126

CURRENT STATUS:

Screenings of materials that have been identified in burley are in progress.

PLANS & TIMETABLE:

Base models for flavor evaluation are being constructed. Evaluations and formulation to be conducted through April, 1983.

IV. Evaluation of Flavoring MaterialsOBJECTIVE:

To evaluate the contribution of flavoring materials to mainstream smoke and pack aroma.

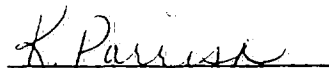
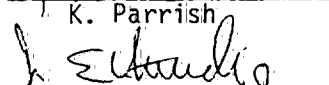
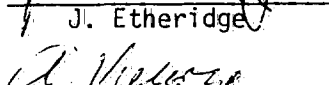
CURRENT STATUS:

The following materials have been evaluated:

- A. Butyric Acid F.E.M.A. - 2221 -sour, earthy, puckery
- B. Baleric Acid F.E.M.A. - 3101 -dirty, more burley
- C. 3 Ethyl Pyradine F.E.M.A. - 3394 -earthy, more burley
- D. 3 Acetyl Pyradine F.E.M.A. - 3324-more burley, horsey

V. References:

Notebook Nos: 7340, 7349, 7658, 7677


K. Parrish

J. Etheridge

A. Vulovic

2022203127

CHARGE NUMBER: 2305
PROGRAM TITLE: FLAVOR DEVELOPMENT - NEW PRODUCTS
PROJECT LEADER: C. N. KOUNNAS
PERIOD COVERED: FEBRUARY, 1983
DATE OF REPORT: MARCH 4, 1983

TITLE: SPECIALTY MENTHOL (PROJECT BINGO I)

WRITTEN BY: B. G. Taylor, W. R. Bell, D. C. Lauranzon
B. G. Taylor *W. R. Bell* *D. C. Lauranzon*

OBJECTIVE:

Develop the best free-standing menthol cigarette.

CURRENT STATUS:

The test market for this project is underway in Rochester, New York. Its non-menthol companion is being test marketed in San Diego, California; Metro New York; Phoenix, Arizona; Portland, Oregon; Atlanta, Georgia; Southern Florida, and Los Angeles, California.

The HTI and POL tests comparing, respectively, the Special Menthol flavor system and the Tobacco Leaf flavor vs. the B&H system are awaiting analytical results before releasing.

The packaging material intended for use for these products does present some organoleptic problems as determined by various aging and storage studies. To minimize or alleviate this problem, the following courses of action have been taken:

- Blend changes proposed
- Application of filter flavors
- Alternative board
- Use of water-emulsion coating instead of present organic solvent coating on board

PLANS AND TIMETABLES:

HTI and POL Testing	(Begin) Early March, 1983
Aging Studies	Continuous
National Introduction of 83's Menthol and Regular	April 14, 1983

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TITLE: PROJECT OTHELLOWRITTEN BY: W. R. Bell, D. C. Lauranzon
W.R. Bell D.C. LauranzonOBJECTIVE:

To develop an 11 mg 83 and 13 mg 100 product with strong appeal to full-flavor smokers.

CURRENT STATUS:

Six prototype models incorporating new blends, aftercut flavors, and filter flavors have been made. These are in the process of being evaluated to determine the best models.

PLANS AND TIMETABLES:

Continue Evaluations and Make Further
Recommendations

March, 1983

Initiate Testing

April, 1983

TITLE: BURLEY FLAVORWRITTEN BY: B. G. Taylor, D. C. Lauranzon
B.G. Taylor D.C. LauranzonOBJECTIVE:

Develop a flavor to be used as an enhancer of burley character in smoke of existing and new brands.

CURRENT STATUS:

The six models that were screened previously have been narrowed down to four. We have received fresh samples of ammoniated bright cigarettes from the Applied Technical Group. These flavors will be applied and evaluated on this cigarette.

A request has been submitted to Semi-works for a 10-lb. run of MF cigarettes with the 10-point reduction in burley with no aftercut flavors added. Also, request for a 10-lb. run of regular Marlboro controls was put in at the same time. Once these cigarettes are available, further screening of the above burley flavors will be done.

POL 3240 was initiated where we will be testing the Marlboro system with a 10-point reduction in burley plus an added burley flavor vs. the regular Marlboro control.

POL 3241 was initiated to give us a baseline where we will be testing the Marlboro system with a 10-point reduction in burley without added burley flavor vs. the regular Marlboro control.

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Both tests will be in the replicated triangle format with panelists consisting of 1026 Marlboro smokers only to determine if a difference can be seen between the two cigarettes.

PLANS AND TIMETABLES:

POL Test

(Begin) March, 1983

Flavor Development to Optimize
Burley Character

Continuous

TITLE: AC Flavor and Casing Substitution Program

WRITTEN BY:

H. Maxwell
*H. Maxwell*OBJECTIVE:

To develop alternative flavor components in order to achieve cost savings, logistical advantages, and guard against shortages.

CURRENT STATUS:

Licorice: Replacement licorice is in test market as Marlboro "B" at 100% utilization. Analytical and subjective screening is ongoing. POL testing of spray dried licorice replacing block licorice, in Marlboro, is in the field. Cigarettes for POL testing of an alternative method for replacement licorice application, in Marlboro and B&H Ultra Lights, were made and are now under analytical evaluation. POL testing in Merit Ultra Lights and Merit is pending cigarette production. Preliminary evaluation of a second method for replacement licorice application is pending cigarette production.

MS: Due to uncertainties in the POL testing results of MS replacement in Virginia Slims Lights and B&H Lights Menthol, incremental utilization by Manufacturing was postponed until these uncertainties are resolved. In order to do this, additional POL testing in B&H, B&H Lights, and Virginia Slims Lights was requested. Cigarettes for these tests have been produced and are now undergoing analytical evaluation. POL testing in Merit Ultra Light is in testing.

Casing
Chocolate: Two candidates for back-up casing chocolate replacement are under consideration:

(A) 1:2 Replacement-Testing is complete.

(B) 1:1 Replacement-POL testing in B&H Menthol (the only test remaining in this program) is pending analytical evaluation of the cigarettes

Vanilla: Candidates for the replacement of 3X vanilla and S-600 are being considered:

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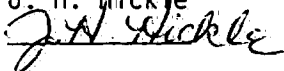
- (A) 3X Vanilla Replacement-Two tests in Merit are complete with satisfactory results. One test, in Merit Menthol, remains incomplete.
- (B) S-600 Replacement-Testing in Merit and Marlboro is complete and acceptable. Testing in Merit Menthol and B&H Lights is incomplete.

PLANS AND TIMETABLES:

Licorice	Monitor Marlboro "B" - Ongoing Complete testing of spray dried licorice - March, 1983 Complete testing of alternative method for replacement licorice application - June, 1983 Evaluate second alternative method for replacement licorice application - March, 1983
MS	Complete testing - June, 1983 Recommend incremental replacement - June, 1983
Casing Chocolate	Shelf Items Complete testing - June, 1983
Vanilla (3X and S-600)	Complete testing - June, 1983 Shelf Items on recommend test market

TITLE: Humectant Study

WRITTEN BY:

J. H. Hickley
OBJECTIVE:

To determine the optimal humectant level for Philip Morris products.

CURRENT STATUS:

The following series of cigarettes was made in the Marlboro configuration with variations of propylene glycol and glycerine levels in the aftercut, casing and burley spray systems:

1. Control - current Marlboro configuration.
2. Test 1 - 50% reduction of propylene glycol and glycerine in all aftercut, casing, and burley spray systems.
3. Test 2 - 100% glycerine elimination in all aftercut, casing and burley spray systems.
4. Test 3 - 100% propylene glycol elimination in all aftercut, casing and, burley spray systems.

Samples of the aftercuts, casings and burley sprays were submitted to the Analytical Research Division for humectant level evaluations. The burley spray

2022203131

Humectant levels are as follows:

<u>Sample</u>	<u>% Propylene Glycol</u>	<u>% Glycerine</u>
(1) Control	*1.27	*1.39
(2) Test 1	0.60	0.80
(3) Test 2	< 0.10	1.40
(4) Test 3	1.20	N.D.

*Adjusted figure due to overdilution with water.

Results of the aftercuts and casings are pending. The cigarette models were submitted to the Cigarette Testing Division for analysis. Results are pending.

PLANS AND TIMETABLES:

Subjective Evaluation

March, 1983

Aging Studies

March/April, 1983

2022203132

CHARGE NO: 2306
PROJECT TITLE: FLAVOR COMPONENT EVALUATION
PROJECT LEADER: R. M. IKEDA
PERIOD COVERED: SEPTEMBER, 1982 - FEBRUARY, 1983
DATE OF REPORT: MARCH 4, 1983
WRITTEN BY: A. M. PALMER

OBJECTIVE:

Apply the Purge and Trap/Gas Chromatographic headspace procedure to the study of flavor related problems.

STATUS:

A. Flavor Loss During Manufacture

To study possible flavor losses during manufacturing, a batch of Marlboro KS filler was sampled at the after-cut flavor cylinder exit, at the silo exit, and from packaged cigarettes. P&T/GC volatiles scans showed a reduction in some components, especially very volatile ones, between the A/C cylinder exit and the silo exit. The major differences, however, were between filler from the silo exit and the filler from packaged cigarettes. There were 20 additional peaks in the packaged cigarette filler which were not present in the silo exit filler, as well as 20 peaks which were smaller and 10 peaks which were larger. Therefore, the packaging and/or adhesives are apparently contributing significantly to the headspace volatiles.

B. Players and Raffles

An extensive study was made of the volatiles of Players and Raffles cigarettes due to a perceived off-taste in these brands. P&T/GC scans were made of the filler of unpackaged Players, Players packed in Players boxes, and of components of the boxes. It was shown that some very large volatiles peaks present in the Players boxed cigarettes are due to the printing on the Players box. These peaks are not present in unpackaged or B&H DUL packaged Players cigarettes. Some extra volatiles peaks were also present in the Players cigarettes packed in B&H DUL boxes, however, these were less intense and in a different part of the scan than those from the Players box. The extra peaks due to the Players box printing were present in both the rods and filters in about equal concentration. It was also shown that these peaks were not greatly reduced in cigarettes from unopened Players packs after 2-3 weeks, so they do not readily dissipate from the unopened boxes. It is obvious, therefore, that packaging volatiles do transfer to the cigarettes and may adversely affect the taste of the cigarettes and must be considered to be an inadvertent additive to the product.

P&T/GC volatiles scans of UK Players cigarette filler and printed box, which is a similarly colored package, also had large peaks in the same area of the GC scan.

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C. Coumarin in Cigarettes and Flavors

Due to the recent popular media interest in coumarin in cigarettes, the P&T/GC volatiles scans made in the last two years of various U.S. brands of cigarettes were reexamined to find those in which coumarin had been detected. A report (1) was issued listing these brands with a rough estimate of the amount present. In addition, four flavors were screened for Operations Services for coumarin content.

D. Miscellaneous Applications

1. Barclay

The taste of U.S. and Swiss made Barclay cigarettes were found to be quite different. The volatiles scans were very different indicating completely different flavor systems.

2. Northwind

The loss of "Creme de Menthe" flavor peaks during accelerated aging was studied by P&T/GC. (2)

3. Cabarrus Merit

P&T/GC volatiles scans were made of a large number of Cabarrus made Merit cigarettes in connection with a possible off-odor problem in the factory start-up.

4. Marlboro - Two Silo/Four Maker/4 Days

P&T/GC volatiles were examined on Marlboro filler sampled from M/C on four different days from two silos and four makers from each silo. There were some intensity differences in about 20 different peaks and this information was reported to Vivian Willis for possible correlation with subjective smoking results. (3)

5. Marlboro - Overheated

Marlboro cigarettes which the filler may have been overheated were examined and the volatiles were slightly less than in the control, but probably not significantly less.

6. P&S Multipass Dryers

Volatiles of Marlboro, Merit and Burley fillers run thru the dryers at 150 and 170°F were compared. With Marlboro filler, there were 25 peaks with different intensities at the two temperatures, most of which were lower at the higher temperature. Merit had 20 and Burley 17 peaks whose intensity varied at the two temperatures, some being more intense and some less intense. Subjective smoking found differences in the Marlboro only.

7. Dunhill Plastic Box

Dunhill boxes containing Merit regular and menthol cigarettes

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were subjected to accelerated aging and the boxes and cigarette fillers examined by P&T/GC. No volatiles from the boxes were detected in the fillers and no significant adsorption of filler volatiles was detected in the boxes. (4) Additionally, four raw materials for making plastic boxes were examined for volatiles content.

PLANS:

1. Do volatiles scans as requested.
2. Further studies of packaging volatiles.
3. Use cryogenic cooling in GC to obtain better resolution and sharper peaks at front end of GC scans.
4. Accumulate GC relative retention time data based on ethyl esters for identification of flavor and tobacco compounds.

REFERENCES:

1. Special Report, Coumarin in U.S. Cigarettes, 2/8/83, A. M. Palmer 83-032
2. Memo from A.M. Palmer to Barbara G. Taylor, Northwind Aging Studies - Purge and Trap/GC Volatiles, 11/22/82
3. Memo from A.M. Palmer to Vivian E. Willis, P&T/GC Volatiles of Two Silo/Four Maker Marlboro Study, 11/23/82
4. Memo from A.M. Palmer to Paula J. Thomas, Dunhill Plastic Boxes, 2/3/82
5. Notebook 7731, pp 111-118



A. M. Palmer

2022203135

CHARGE NUMBER: 2307
PROGRAM TITLE: FLAVOR DEVELOPMENT - TOBACCO MATERIALS
PROJECT LEADER: J. W. SWAIN
PERIOD COVERED: FEBRUARY, 1983
DATE OF REPORT: MARCH 7, 1983

I. RL Flavor Development

A. Pilot Plant RL Evaluation (R. Mitchell) *R. Mitchell*

OBJECTIVE:

Subjectively evaluate and recommend further testing of various Pilot Plant RL's.

STATUS:

Screening of 100% and 27% Bridge RL cigarettes has shown Abatibi Kraft to be less different from the RL-TC control than Domtar Kraft. However, the Bridge RL-TC with 6.8% Abatibi Kraft which was the preferred level was slightly different from the control.

Coated RL's from the Pilot Plant program testing different process conditions have been received for subjective evaluation.

PLANS:

Evaluation of lower levels of Abatibi Kraft has been scheduled.

Subjective evaluations of Pilot Plant coated compared to sized RL-TC has been initiated with 17, 27, and 100% RL-TC cigarettes. Park 500 RL's from control and test (coated) blends will also be evaluated in March.

B. Evaluation of Y Test Market Marlboro (V. Willis) *V. Willis*

OBJECTIVES:

1. Determine if differences exist among control, Y, and F Marlboro cigarettes.
2. Determine if differences exist between aging and subjectives during aging processes.
3. Initiate Y-84TM evaluations and monitor Y production through freezer studies.

STATUS:

Accelerated aging of January production of Y-84TM samples with CTB has been completed.

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MARCH 7, 1983

Analytical results for comparison to previous blends are incomplete. Subjective evaluation continues on equilibrated and unequilibrated cigarettes from hot-wet, hot-dry, and accelerated aging conditions. Initial evaluation of February production has been completed.

PLANS:

Subjective and analytical results will be compared for Test Market and current production Marlboro blends. Freezer storage evaluations will continue to compare blend changes.

II. ET-NH₃ Control (W. Stephens)OBJECTIVE:

To evaluate the subjective and physical effects of treatment during reordering.

STATUS:

Y82-2 and Y84-1 Marlboro blends with 25% levels of aged ET, DET, and ET Test 3 (Production Trial 2) were subjectively evaluated to verify differences found between fresh ET and DET in the Y82-2 blend. Results indicated no significant differences among the aged (6 months) expanded samples in either blend. Therefore, an aging influence on the NH₃ flavor effect of ET has been confirmed. Chemical analyses of aged materials are in progress.

PLANS:

Complete consumer panel testing (N-3220) and recommend further production tests to evaluate physical and subjective effects in current blends.

III. REFERENCES

1. Notebook #7614
2. Notebook #7657
3. Notebook #7710
4. Notebook #7736



2022203137

CHARGE NUMBER: 2308
PROGRAM TITLE: ROOM AROMA COMPONENT EVALUATION
PERIOD COVERED: February, 1983
PROJECT LEADER: A. G. KALLIANOS
DATE OF REPORT: MARCH 9, 1983
WRITTEN BY: A. G. KALLIANOS

I. Sidestream Smoke Studies

OBJECTIVES:

1. Identify undesirable components in sidestream smoke and effect compositional changes to reduce them.
2. Develop the technology to reduce quantity or visibility of sidestream smoke.
3. Develop the technology to reduce or mask objectionable odor of sidestream smoke.

STATUS:

A. Sensory evaluation of sidestream smoke (B. Demian)

1. The descriptive sensory data for the full-flavor group of the twenty-seven commercial brands selected for study were statistically analyzed with the assistance of Analytical Research personnel (M. Jeltma and B. Good). Analysis of variance is showing that the panelists are consistent in their ratings, but there are differences in discriminative powers. The panel as a whole can distinguish between the ten "full-flavor" brands on basis of sidestream smoke odor, i.e. "ammonia", "pyrazine", and to some extent "smoky" attributes. Higher than average ratings on these three descriptors were obtained for the control A cigarette, manufactured from a commercial blend of tobaccos. Raleigh, Viceroy, and Control C were judged lower than average in "pyrazine" notes. Pall Mall sidestream smoke is much higher in "smoky" character.
2. We are currently in the process of evaluating the seven brands of commercial cigarettes grouped in the "Lights" category. Initial screening for differences is being conducted with triangle tests.
3. A competitor test cigarette, being sampled in a Mall study with some questions relating to sidestream smoke, was evaluated in a descriptive analysis by our sidestream smoke panel. The test cigarette is mentholated and is fabricated with a high citrate double wrapper. The panel found it to be very high in smoky-burnt attributes and very harsh. Same attributes but of smaller amplitude were observed for a high citrate, non-porous wrapper experimental cigarette.
4. The sidestream smoke evaluation apparatus is now fitted for sample collection and awaiting test results from Analytical Research personnel on the "Chrompak".

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thermal desorption-Cryogenic trapping unit.

B. Control of sidestream smoke

1. Cigarettes of a commercial brand extracted with a variety of solvents have been submitted to descriptive sensory analysis of sidestream smoke. The most characteristic change observed in extracted cigarettes is an increase in "smoky" and "burnt" attributes, and this increase parallels the weight change after extraction. The higher the weight change, the higher the smoky-burnt character.
2. Exploratory work to modify the odor of sidestream smoke has been continued. An alcoholic extract of tarragon was found to increase the "sweet" character of sidestream smoke.
3. Experiments to mask/neutralize the butt aroma of cigarettes have involved a variety of masking flavorants and odor control chemicals. Volatile acids and alcohols appear promising.

II. Burley Tobacco Character (with H. Lanzillotti)

OBJECTIVE:

Explore the development of burley tobacco character on RL.

Our approach in meeting this objective is based on oxidative and deaminative reactions that appear to take place in burley tobacco during the air-curing process. Initial experiments have focused on the oxidative reactions and these are being explored through ortho-quinone formation of polyphenols and oxidation of ascorbic acid. Both of these appear promising in developing desirable aroma and flavor on RL. Our experiments with oxidized ascorbic acid, however, produced conflicting results based on expectations from a large body of literature on the structure of dehydroascorbic acid. C^{13} -NMR spectra by Dr. Richard Cox confirmed our observations and are helping our better understanding of the reactions involved.

Experiments to resolve conflicting information through study of derivatives are in progress, as are also experiments to optimize formation of flavor intermediates and use levels on RL as well as on tobacco blends.

III. References

1. A. G. Kallianos, Notebook #7619
2. B. Demian, Notebook #7849
3. H. V. Lanzillotti, Notebook #7709


A. G. Kallianos

2022203139

CHARGE NUMBER: 2501
PROJECT TITLE: Nuclear and Radiochemistry of Smoke
PROJECT LEADER: Roger A. Comes
PERIOD COVERED: February 1-28, 1983
DATE OF REPORT: March 7, 1983

Low Level Laboratory

Samples of "class tobaccos" that had been counted on the low background alpha-beta counting system were re-counted during this report period. Beta activity only is listed. Class tobacco types are described in Project 2501 Annual Report-1982, Acc. #83-029. The consistency in the data between the two dates (9 months apart) enhances the faith one can put in the instrumentation. Drift over this time period has been minimal with the Tennelec system.

<u>Class Tobacco</u>	<u>1/83 (pCi/g)</u>	<u>4/82 (pCi/g)</u>	<u>Δ(pCi/g)</u>
1	25.9	24.7	+1.2
2	28.8	28.4	+0.4
3	24.5	29.2	-4.7
4	40.7	40.2	+0.5
5	46.9	43.5	+3.4
6	27.4	27.7	-0.3
7	50.0	49.3	+0.7
8	25.7	27.5	-1.8
Control	26.1	25.0	+1.1

These class tobacco materials are being utilized additionally to study other counting effects, i.e. dust vs granular samples and thin film vs thick layered samples.

Health Physics

Surveys were conducted in laboratories assigned to Projects 2501, 2525, 2506, 6910 and 6902. Wipe tests were carried out on sealed sources in the Semi-works. Health physics aspects relating to the use of ^{32}P by the Biochemistry Division continue.

Capillary Chromatography

An alkaloid containing fraction of smoke was analyzed for the presence of nicotine at the request of Project 2525. The sample was found to contain 20ng/ μl nicotine with a quantitative variation of less than 10% and a retention time variation of 0.3%.

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A procedure was established for analysis of TMS derivatives of abienol. One sample was analyzed for Project 2525.

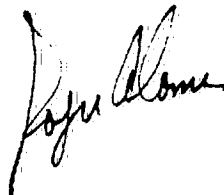
Mainstream smoke fractions from VdC smoking run #2 were analyzed using both NPD and FID. In addition, mainstream and sidestream fractions from 2R1 and Monitor-22 smoke runs were evaluated.

^{14}C -Nicotine Cigarettes

The first batch of cigarettes containing ^{14}C -nicotine (Code 2-83-1) machine made by Project 2506 for use in the VdC project were sectioned for uniformity of labelling and smoked for total distribution. The section data showed excellent distribution of radioactivity throughout the rod with an average specific activity of $11.0\mu\text{Ci/g}$ tobacco. The total smoke distribution data are listed along with data from hand-made ^{14}C -nicotine cigarettes (Code 11-3-82) smoked earlier. Both cigarette types contain 65mm of the "VdC blend" in Schweitzer 156 paper and have #2051 whistle thru filters attached.

^{14}C -Nicotine Cigarettes
TOTAL SMOKE DISTRIBUTION (%)
(Five Run Averages)

	<u>Code #2-83-1</u>	<u>Code #11-3-82</u>
SS: Gas	15.4	15.7
MS: Gas	11.1	1.2
SSTPM	46.3	37.9
MSTPM	18.3	16.6
Butt (filler)	10.4	22.6
Butt (filter)	8.2	6.0
Ash	0.3	0.2



2022203141

CHARGE NUMBER: 2506
PROJECT TITLE: Botanical Investigations
PROJECT LEADER: Roger T. Bass
PERIOD COVERED: February 1-28, 1983
DATE OF REPORT: March 10, 1983

I. GENERAL

A. The roots of the ^{14}C tobacco plants from VdC chamber runs No. 3 and 4 have been completely air dried and cleaned resulting in the yields shown below. Specific activity determinations for this material are being made.

<u>VdC Run No. 4</u> <u>Variety</u>	<u>Plant No.</u>	<u>Root Yield (Dry Wt.)</u>
Ky. 10	1	34 gms.
Ky. 10	2	56
Ky. 10	3	42
Ky. 10	4	64
	Total	196

<u>VdC Run No. 3</u>		
S. C. 58	1	46
S. C. 58	2	40
S. C. 58	3	54
S. C. 58	4	34
	Total	174

B. A ten gram quantity of sieved Ky. Ref. 2R1 filler was sprayed with a 3ml. volume of duvatriediol solution. The DeVilbiss No. 15 atomizer was used for the application. After equilibration, a total of eight hand made cigarettes were prepared for use in smoke studies by Project 2501.

C. At the request of Project 1720 a quantity of C-319 tobacco leaves were flue-cured in the oven and then equilibrated for shredding. Another group of harvested leaves were sun dried on the Greenhouse bench for utilization in expansion studies. The air curing of several groups of leaf samples were also completed.

D. Artificial plant light intensity measurements were made and information provided for the optimum ratio and balance of fluorescent and incandescent light sources being utilized by Project 6902 in their incubator cabinet.

E. On 1 February the ^{14}C -nicotine VdC cigarette making operation was successfully completed on the new Hauni Baby making machine, under the supervision of the Radiation Safety Officer. In a short period of time we produced a total of about two thousand ^{14}C -nicotine labelled cigarettes to be utilized in VdC smoke

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studies. About one half of these were 85mm non-filter cigarettes and the other half were 65mm cigarettes to which we will attach "whistle through" cellulose acetate filters. Preliminary determinations on these cigarettes indicate an average weight and RTD as shown below.

	<u>65mm (w/filter)</u>	<u>85mm (no filter)</u>
Avg. Weight	0.97gm	1.24gm
Avg. RTD	2.86 in. H ₂ O	2.50 in. H ₂ O

This achievement, the successful preparation of these cigarettes, marks the culmination of planning, plant selection, tobacco production, ¹⁴C-nicotine isolation, and labelled nicotine application to filler, which all began over two years ago for the cooperative VdC study.

II. COOPERATIVE STUDIES

A. The 1982 Kentucky Burley Regional Farm Test tobacco variety display was attended in Lexington, Ky. at Penn Brothers Warehouse. The displayed tobaccos were evaluated in cooperation with Leaf Department personnel as to their physical characteristics as reflected in their suitability for PM use (or % graded as usable). For the Regional Farm Test, the two control varieties Ky. 14 and Bu. 37 were compared to the three experimental entries Coker 80-42E, Gr. 115, and Rogers 80-63-14. The percent graded by PM for each entry was as follows: Ky. 14 (80%), Bu. 37 (81%), Coker 80-42E (96%), Gr. 115 (80%), and Rogers 80-63-14 (74%). For the Regional Small Plot Test at the Spindletop, Ky. location all of these same five entries showed excellent appearance with 100% graded usable for each of them. Burley tobacco from several other tests, including the Sucker Control Test, and the Potassium Source Test (KCl and K₂SO₄ applied in the spring and fall at 100, 200, 300, and 400 pounds per acre rate) were also evaluated.

B. Cigarette samples from 1982 Burley Tobacco Regional Farm Test were received and evaluated by the Subjective Screening Panel. The experimental entries (Coker 80-42E, Gr. 115, and Rogers 80-63-14) were compared to the control varieties Ky. 14 and Bu. 37 and were found to be acceptable in burley smoke flavor characteristics. This information was reported to the Variety Evaluation Committee.

III. GREENHOUSE

A. Because of the interest in cis-abienol, a leaf surface lipid, we have obtained seed of the Red Russian variety and some plants will be grown for use by L. Weissbecker.

B. Fresh green leaves have been provided as requested from plants of C-319, N. sylvestris and N. tomentosiformis for cis-abienol determinations by Project 2525. Various other tobacco material samples have been supplied as requested for Project Nos. 1720, 1730, 1754, 2525, 2501, and 6902.

C. Greenhouse maintenance includes the replacement of several glass panes and the installation of new Stuppy watering tips on the automatic watering system. These tips are an improvement over the original ones, which often stopped up.

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D. The normal seeding and transplanting activities have been completed, including Wisconsin 38, Ky. 14, Coker 319, and Oriental varieties.

References:

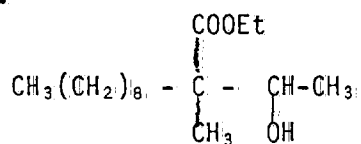
A. Brna, N. B. 7806
G. Newell, N. B. 7295

Roger T. Bass

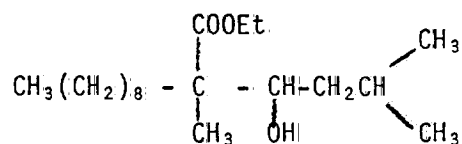
2022203144

CHARGE NUMBER: 2515
 PROJECT TITLE: Flavor-Release Chemistry
 PROJECT LEADER: Yoram Houminer
 PERIOD COVERED: February 1-28, 1983
 DATE OF REPORT: March 9, 1983

We are continuing our research in the area of aldehyde release. Ethyl 2-(1-hydroxyethyl)-2-methylundecanoate (I) was prepared in 59% yield by reacting the enolate anion of ethyl 2-methylundecanoate with acetaldehyde.¹ The hydroxy-ester derivative of isovaleraldehyde (II) was obtained by a similar procedure.² In both cases, the products are mixtures of two diastereoisomers in about 1:1 ratio.



I

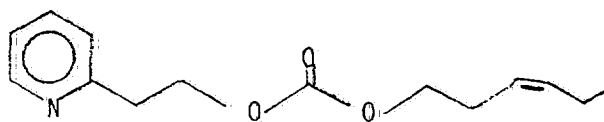


II

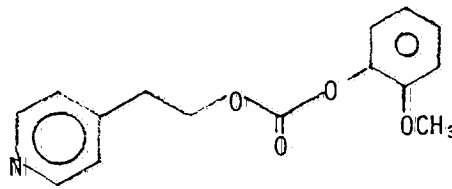
Pyrolysis of I at 300°C for 1 min. under a flow of helium yielded 18% of acetaldehyde. At 400°C, a quantitative yield of acetaldehyde was obtained.³ Pyrolysis of II at 250°C for 5 min. in an open tube gave 45% release of isovaleraldehyde. We plan to further explore the potential of these systems as precursors for low molecular weight aldehydes.

All eight possible isomers of phenyl aldopentopyranosides (the α and β -anomers of arabino-, lyxo-, ribo- and xylopyranosides) were purified.¹ Conformational analysis by NMR will be carried out, and we hope that such information will provide insight on the mechanism of the aryl glycosidation reaction.

We are continuing our study in the area of carbonates derived from pyridyl-ethanols and a variety of phenols and alcohols. Compound III and IV were prepared by reacting the corresponding pyridylethanol with the corresponding chloroformate.⁴



III

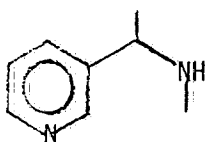


IV

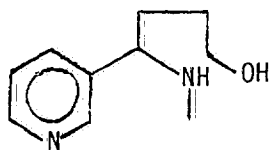
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Pyrolysis of III at 250°C for 10 min. gave only 5% of the desired *cis*-3-hexenol, whereas the phenyl analogue of III has been shown to give >50% of phenol under similar conditions. These results indicate that carbonates derived from 2-pyridylethanol are good precursors for phenolic flavorants but not for alcohols.⁴ Pyrolysis of carbonates of the 4-pyridyl isomer will be carried out in the near future.

We have successfully prepared the amine V by reacting 3-acetylpyridine with methylamine in the presence of NaCNBH₃. This methodology will be applied in the preparation of VI, a precursor designed to release nicotine.⁴



V



VI

We have completed the analysis of levels of CR-1845 on cigarettes that were fabricated for smoking studies. Our five-port smoking machine was recently refurbished and preliminary smoking experiments are now in progress.⁵

A patent disclosure entitled "Dicarbonates of 2,5-dihydroxy-1,4-dioxane as release agents for alcohol and phenol flavorants" by K. F. Podraza was submitted to the patent department. A manuscript entitled "Stereoselective synthesis of aryl aldopyranosides" was submitted to MRB by G. Chan.

References

1. G. Chan, 7734
2. Y. Houminer, 7747
3. Carried out by L. Brown
4. K. Podraza, 7851
5. J. Kang, 7692

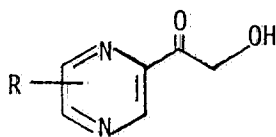
Yoram Houminer

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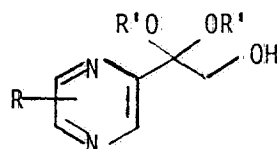
CHARGE NUMBER: 2520
 PROJECT TITLE: Synthesis of Flavorants
 PROJECT LEADER: William B. Edwards, III
 PERIOD COVERED: February 1-28, 1983
 DATE OF REPORT: March 7, 1983

I. Heteroaromatic Flavorants

A mixture of 2-oxo-2-(5- and 6-methylpyrazinyl)ethanol (I and II, 1:10 ratio) was synthesized in two steps from 2-acetyl-5- and 6-methylpyrazine via the dimethoxyketals (III and IV). Preliminary GC/MS analysis strongly indicated that I and II are pyrolysis products of 2,5- and 2,6-deoxyfructosazine, respectively.¹ Initial attempts to prepare I and II by hydrolysis of the diethoxyketals (V and VI) yielded only a trace amount of the desired products. Additionally, 2-acetyl-3-ethylpyrazine was hydroxylated to give the diethoxyketal (VII). Hydrolysis of VII to the corresponding hydroxyketone will be attempted. Compounds I through VII will be evaluated for their cigarette flavor properties. All had no appreciable odor at room temperature.²



I, R = 5-CH₃
 II, R = 6-CH₃



III, R = 5-CH₃, R' = CH₃
 IV, R = 6-CH₃, R' = CH₃
 V, R = 5-CH₃, R' = C₂H₅
 VI, R = 6-CH₃, R' = C₂H₅
 VII, R = 3-C₂H₅, R' = C₂H₅

The following potential flavorants were placed in the CR file.³

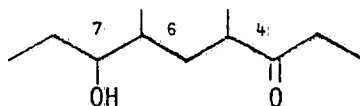
CR-2245 - 2-Acetoxy-6-methylpyrazine
 CR-2278 - 2-Acetoxy-3-methylpyrazine
 CR-2279 - Isoquinoline N-oxide

II. Entomological Research

Initial equilibration studies carried out on (+)-serricornin (VIII) showed no change in isomer ratio detectable by NMR analysis.⁴ This result strongly indicates that isolated VIII is fully equilibrated at the 4 carbon. While it has been

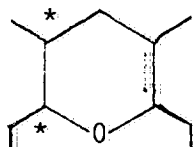
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reported by others that VIII is readily epimerized at the 4 position, this needs to be confirmed by us in order to complete the development of a stereocontrolled synthesis of VIII.²



VIII

A method for the large scale preparation of anhydroserricornin has been established. Compound IX can be synthesized in 77% yield by acid catalyzed dehydration of (+)-serricornin (VIII) in the appropriate solvent. These results far exceed existing literature methods which gave IX in about 25% yield. Compound IX is obtained as a mixture of two diastereomers in a 2:1 ratio. The chiral centers (*) of IX correspond to the 6 and 7 carbons of VIII. The isomer ratio at carbons 6 and 7 is reproducibly fixed in an earlier synthetic step and should not be altered by the subsequent chemistry. Consequently, our method should consistently yield IX with the same diastereomer ratio.²



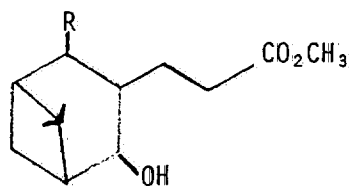
IX

The two step synthesis of 2,4-dimethyl-5-oxoheptylnitrile (X) developed last month was carried out on a 2.3 molar scale. Little variation in reaction yield was noted. Isomer equilibration/reduction of X gave 5-hydroxy-2,4-dimethylheptylnitrile (XI) with the same diastereomer ratio as previously obtained. Using liquid chromatography, XI was separated into two isomer fractions. Analysis of the fractions by nuclear magnetic resonance⁴ showed that one was a single diastereomer of XI while the other was a mixture of three isomers.²

III. Terpenoid Flavorants

Cembrenoid Flavorants: Reduction of methyl 3-[3-(6,6-dimethyl-2-oxobicyclo[3.1.1]heptyl)]propionate (XII) and methyl 3-[3-(4,6,6-trimethyl-2-oxobicyclo[3.1.1]heptyl)]propionate (XIII) gave the corresponding alcohols (XIV and XV). These alcohols will be evaluated for their cigarette flavor properties.³

2022203148



XIV, R = H

XV, R = CH₃

1. D. Ingraham and J. Naworal
2. D. Williams: N.B. 7847
3. C. R. Howe: N.B. 7707
4. R. Cox

William B. Edwards

2022203149

CHARGE NUMBER: 2525
 PROJECT TITLE: Chemistry and Isolation of Tobacco Constituents
 PROJECT LEADER: Harvey J. Grubbs
 WRITTEN BY: Richard R. Izac
 PERIOD COVERED: February 1-28, 1983
 DATE OF REPORT: March 14, 1983

Cigarettes made from four different blends (See Table) have been smoked on a 30 port Borgwaldt smoking machine.¹ The sidestream (SS) obtained from the electrostatic precipitator and the mainstream (MS) obtained from a standard Elmenhorst trap were fractionated to obtain the base fraction.² An attempt is being made to define the differences between the various base fractions.³

Cigarette	Number Smoked	Condensate(g)	Base Fraction (g)
2R1	119		
SS		4.7	0.5215
MS		4.5*	0.3705
100% bright	120		
SS		6.4	0.9378
MS		6.7*	0.8459
100% burley	115		
SS		5.8	0.8283
MS		4.3*	0.7145
100% Oriental	120		
SS		7.8	0.4691
MS		5.1*	0.2968

* Contains any water trapped

Using 117 cigarettes spiked with ¹⁴C-(U)-nicotine tartrate (1.62μCi/cig.),⁴ a second VdC smoking run has been completed.^{1,2,5} The sidestream condensate (23μCi) obtained from the electrostatic precipitator and the mainstream condensate (25.4μCi) obtained from the Elmenhorst trap have been fractionated by a modified Snook procedure to obtain a nicotine free aza-arene fraction. Results indicate that the aza-arene fraction accounted for about 0.5% of the activity found in the sidestream⁴ and about 0.4% of the activity found in the mainstream. Preliminary analysis of the mainstream fraction indicates that the major component, nicotine, is about 20% of the fraction and as much as 30% of the activity.^{2,6}

Citrus seeds obtained from Ventura Coastal Corporation have been extracted with acetone and the extract defatted to yield an enriched limonoid fraction (0.3% of the seed weight).² This fraction as well as pure limonin⁷ are being evaluated as antifeedants by S. Long (Project 1101).

2022203150

A presentation entitled "Cigarette Smoke and Aerosol Production Research at Philip Morris" was given at the University of Virginia by Doug McRae.¹

References:

1. D. D. McRae, Notebook #7884
2. R. R. Izac, Notebook #7841
3. D. F. Ingraham, Notebook #7699
4. These were 85mm filterless cigarettes and were prepared on the Hauni Baby by Project 2506.
5. M. Core, Notebook #7749
6. F. Greene, Project 2501
7. Analyzed by J. Brady, Notebook #7868

R. Izac

2022203151

CHARGE NUMBER: 4009

PROJECT TITLE: Development Smoke Studies

PROJECT LEADER: B. L. Goodman

PERIOD COVERED: February, 1983

I. PROJECT SLOW (K. Gunst)

Objectives:

Determine the effect of cigarette components on sidestream visibility.

Develop a reduced sidestream product that is subjectively acceptable.

Status:

Measurements of light extinction gave similar values for a nonporous control cigarette wrapper and one with potassium acetate coating. The coating reduced the static burn time and gave a 25% reduction in sidestream TPM. The appearance of the ash was also greatly enhanced.

Glutaric acid on the wrapper gave a 10% reduction in the amount of light extinguished compared to a nonporous control. TPM reduction was 20% at an equivalent static burn time.

Citric acid gave reductions in both visibility and TPM, but the level was not significant compared to the nonporous control. Nonporous wrappers have previously shown to reduce the visibility of sidestream smoke by 30-35% without any coating.

Plans:

To coat wrapper with potassium glutarate for potential reduction of sidestream TPM.

Measure sidestream visibility and tar on selected commercial brands and experimental cigarettes.

Design an optimum model with reduced sidestream and acceptable taste, odor and delivery.

II. COMPUTER APPLICATIONS (R. Greene)

Objective:

To write programs in BASIC and provide user assistance for the directorate.

Results:

Two library directories have been created for the Cigarette Development Division and the Flavor Development Division. These libraries are open to computer users in either division and provide sample command files, demonstrations for the novice user and various automatic programs to facilitate computer tasks. Most of the computer directories in the division have been modified to access this library.

Several small programs were written and added to the BASIC library. They include a file reformatting program to transform column data files and output raw data files, several upgrades to "BASIC-PLUS-2 to EASYGRAPH UTILITY ROUTINES" and creation of additional string array processing subprograms. Most of the changes were implemented in

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routine maintenance of the large programs delivery, onecurve, rate, propburn, math-pack and tipanal. The onecurve program has been updated to allow data merging of several file sections for additional statistical analysis.

An APL function has been written by Dr. Hartung to allow file communication between delivery and the APL modeling package.

III. SMOKER SIMULATOR PROGRAM (M. Kelley, R. Arthur)

Objective:

To evaluate, update, and utilize Human Simulator equipment.

Status:

The new four port Simulator has been installed in its permanent location. Preparation of command tapes or discs with the input of human smoking data has given the desired data. That portion of the program is now in the process of being converted to the DEC by CAD personnel.

Reproducibility of the puff volume has given some problems. The testing indicated that the thickness of the Cambridge pad is critical. If it is less than 1.000" thick, there is loss of volume, and if it is more than 1.014" there is binding in the transfer mechanism. New springs have been ordered.

The built-in puff selections were reprogrammed to permit variable puff intervals. They can now be set at 35 seconds or longer by the operator.


The single port machine was used for smoking experimental cigarettes for nicotine changes due to various treatments of the tobacco. The cigarettes were made by P. Martin's project and the results were forwarded to them.

Another short study of differences in smoking profiles between a control cigarette and a new product was completed using the PPA to measure puff volumes and durations. No differences in profiles were seen for the eleven panelists.

Plans:

To complete the evaluation of new equipment and install modified parts.

Complete the conversion of all Simulator functions to the DEC.


B. Goodman

BG/lad

2022203153

CHARGE NUMBER: 4010
PROJECT TITLE: Brand Modifications
PROJECT LEADER: W. A. Geiszler
PERIOD COVERED: February, 1983

I. SMOKE DELIVERY CHANGES

A. Marlboro (L. Stewart)

Objective:

Determine consumer acceptability of Camel Filter KS versus Marlboro KS.

Status/Plans:

A POL test comparing Marlboro (16 mg FTC tar) versus Camel Filter (14 mg) is in the field.

B. Marlboro for Saudi Arabia (L. Stewart)

Objective:

Develop versions of Marlboro LS, KS and 100's for export that do not exceed 15.0 mg tar/1.0 mg nicotine deliveries using the ISO smoking method.

Status:

Models of Marlboro LS, KS and 100's with 20% ET in the blend have met the tar and nicotine delivery requirements, and have performed well in internal panel testing. Complex Panel testing is being conducted on the KS and 100 mm versions. Models with 6% glycerine on the filter (and the conventional Marlboro blend) have met the nicotine delivery requirement when fresh, but the nicotine delivery increases after aging.

Plans:

Complete Complex Panel testing of the 20% ET models and conduct mailout testing if warranted.

C. Cambridge (J. Nepomuceno)

Objective:

Develop an improved version of the 1 mg 84 mm Cambridge product.

Status/Plans:

Cigarettes with flavored $Mg(OH)_2$ granules in the filter are being tested by the discriminative panel after six weeks of aging in the pack. Subsequent testing by the Complex Panel is planned.

D. Marlboro Lights (E. Stagg)

Objective:

Develop a 9 mg FTC tar version of Marlboro Lights KS that achieves parity with the 11 mg current product.

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Status/Plans:

A 9 mg KS model with a modified flavor formulation is being tested by the Complex Panel. Another model with a modified blend is being made for Complex Panel testing.

II. CIGARETTE TECHNOLOGYA. Filter Flare-Up (K. Gunst)Objective:

Develop a white tipping with 0% filter flare-up rate in machine smoking and $\geq 88\%$ opacity.

Status:

A GSR-156 basesheet modified with monoammonium phosphate by Kimberly-Clark was evaluated as a candidate for meeting the opacity and flare-up requirements. In contrast to the results obtained on previous samples, the MAP-modified sheet gave no reduction in flare-up rate. Cigarettes are being made to re-test this sheet.

Extensive testing of Project Bingo cigarettes, which use a cork-on-white tipping, has been conducted to assess filter flare-up potential. The 100 mm cigarettes have given 0% flare-up, but the 83 mm product has given a 3% flare-up rate. Since the 100 mm tipping has a longer white skirt than the 83 mm tipping, tippings with various skirt widths have been ordered for testing on the KS product.

Plans:

Continue evaluation of new and modified white tippings.

B. Plugwrap Evaluations (E. Stagg)Objective:

Evaluate new or modified plugwrap materials.

Status/Plans:

Three new plugwrap materials are being evaluated: (1) a 4,500 CORESTA porous plugwrap from Dexter; (2) a 16,500 CORESTA porous plugwrap from Kimberly-Clark; and (3) a Parliament mouthpiece material from Colonial Heights.



W. A. Geiszler

WAG/lad

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CHARGE NUMBER: 6902
PROJECT TITLE: BIOCHEMICAL SPECIAL INVESTIGATIONS
PERIOD COVERED: February 1-28, 1983
PROJECT LEADER: T. Yu
DATE OF REPORT: March 3, 1983

1. V79 SISTER CHROMATID EXCHANGE ASSAY¹

A four-hour treatment with the direct acting positive control compound ethylmethanesulfonate (EMS) induced a dose related increase in sister chromatid exchanges (SCEs) in V79 cells. SCEs per metaphase chromosome were: 7.5 ± 2.0 , 11.3 ± 2.8 , 19.5 ± 4.7 , 17.6 ± 2.1 and 27.7 ± 6.7 at 0, 0.3, 0.6, 1.0, and 3.0 mM, respectively. No second division cells were observed at the highest dose tested, i.e., 10 mM. A replicate experiment has been conducted and the SCEs are being scored currently. These results will be evaluated to determine interexperimental variations.

2. ³²P-POSTLABELING ASSAY²

New shipments of ³²P-ATP and DNA digestion enzymes were received this month. These materials were used for digestion and phosphorylation reactions. As a quality control measure, the ³²P-ATP was chromatographed and autoradiographed (TLC-AR) upon arrival and subsequently at time intervals throughout the month. The TLC-AR results showed that ³²P-ATP contained a major highly radioactive spot and five additional spots. The RF value of the major spot was comparable to that of the authentic ATP. The radioactivity of the additional spots as reflected by the intensity of the spots increased with increasing storage time. Five replicate phosphorylation reactions were carried out using authentic nucleotide monophosphates. The results indicated that the reaction was proceeding and the impurities in ³²P-ATP did not interfere with the reaction. Methods and procedures on quantitation of radioactivity by Cerenkov counting is being worked out in cooperation with Project 2501 personnel.

Two experiments were carried out on the digestion of calf thymus DNA and the subsequent phosphorylation with ³²P-ATP. The digestive enzymes were dialyzed overnight. The results showed that both the digestion and phosphorylation reactions proceeded.

Work will proceed to finish assay establishment by using positive control compounds.

3. NEUROCHEMISTRY (with Project 1610)³

Two experiments were carried out in this area.

4. TRADESCANTIA STAMEN HAIR MUTATION ASSAY⁴

A visit was made to Brookhaven National Laboratory to learn the methodology of this assay system. Cuttings and potted plants were brought back and one experiment is currently in progress.

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5. REFERENCES

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Terry Yu

nwp

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CHARGE NUMBER: 6904

PROGRAM TITLE: BIOCHEMICAL METHODS DEVELOPMENT AND UTILIZATION

PERIOD COVERED: February 1-28, 1983

PROJECT LEADER: R. W. McCuen

DATE OF REPORT: March 1, 1983

I. V79 CHINESE HAMSTER CELL INHIBITION OF METABOLIC COOPERATION (IMC) ASSAY

Several new chemicals were tested in an attempt to more fully understand the nature of activity in this assay. Two experiments^{1,2} were completed using various doses of ethanol which, through personal communication with another investigator using this system, should not be active. Our data indicates that ethanol is weakly active, however variability precludes any definitive statement. This agent will not be retested if one of the other chemicals currently being examined prove to be a suitable negative.

An unknown pure chemical was submitted for testing. Due to its acid characteristics, the pH of the cell culture medium had to be adjusted using sodium hydroxide. Results were obtained which suggested that while the agent was slightly toxic to the V79 cells (about 25% killing at the 1-2 mg/ml doses), it did not block metabolic cooperation (*i.e.*, was negative).³ Studies are currently underway to confirm and extend these results.

A second experiment was done using dry IT 2R1 CSC to confirm earlier findings.⁴ As before, the condensate appeared weakly active (1.6 X background) at about 10 µg/ml.² The strong toxicity that was observed tended to compromise this conclusion. More tests with this CSC and at least one other condensate (LTF-5E; nitrogen-free filler) are planned to determine if the pattern of weak activity and high toxicity are a common characteristic of CSCs in this assay.

II. L5178Y TRIFLUOROTHYMININE RESISTANCE ASSAY³

A new microsomal (S9) preparation has been tested and found to be satisfactory for our needs. A memo detailing these results has been written. As indicated previously,⁵ no further investigations are planned with this assay due to the increased effort with the IMC system.

III. REFERENCES

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3. Ayers, D. J. Notebook No. 7839, p. 75.
4. Penn, J. M. Notebook No. 7773, pp. 149-156.
5. McCuen, R. W. 6904 Monthly progress report. Monthly Progress Report 83-031. 1983 February 15.

E. W. McCuen

CHARGE NO. 6906
PROJECT TITLE: BIOLOGICAL EFFECTS OF SMOKE
PERIOD COVERED: February 1-28, 1983
WRITTEN BY: M. H. Tickle
PROJECT LEADER: K. Rapp Sherwood
DATE OF REPORT: March 9, 1983

I. SALMONELLA/MICROSOME (S/M) MUTATION ASSAY--CHEMICAL DETERMINANTS OF IT CSC ACTIVITY (with 6908)

One of the continuing goals of the collaborative studies with Project 6908 has been to isolate and identify certain compounds which are responsible for impaction trap cigarette smoke condensate (IT CSC) activity. Two compounds, MW 270 and MW 211, which were first seen in the burley base fraction and then later reisolated by reverse phase liquid chromatography (RPLC) from LH-20 fraction 8, were tested using strain TA98 with metabolic activation (+S9). Also tested were LH-20 fraction 7 and its six RPLC subfractions.¹ The results indicated that MW 270 and MW 211 were both active. LH-20 fraction 7 and each of its six subfractions also showed activity.

II. S/M ASSAY (STRAIN TA98)--ASSAY RESPONSE USING A MODIFIED FORWARD MUTATION ASSAY PROTOCOL

Previous experiments to include a measure of toxicity as part of the Ames *Salmonella typhimurium* assay, using the protocol of the 8-azaguanine forward mutation assay, were not successful. A modified version of this protocol was then designed and tested with 2R1 CSC.² In a similar manner as the forward mutation protocol, the modified protocol was designed to quantitatively test for both the number of revertants (mutation) and the number of survivors (toxicity) using the same exposed cell population. The effect of different incubation times (0, 30, 60, and 120 minutes) for the reaction mixtures were tested at each 2R1 dose. Aside from trying to mimic the two hour incubation time used for the 8-azaguanine assay, the purpose for testing these various incubation times was to determine if the incubation time would affect the observed response using TA98.

The data indicate that the number of revertants increased at all doses as the incubation time increased. This increase was most evident for the one and two hour incubation times, along with evidence of toxicity especially at the CSC dose of 1.0 mg. Using the toxicity data, the mutation frequency (number of revertants per exposed cell population) was calculated. Future studies will be conducted to determine what type of toxicity determination, if any, could be included as a modification to the Ames assay protocol.

III. S/M ASSAY--SIDESTREAM AND MAINSTREAM SMOKE STUDIES (with 6910)

In our continuing studies with sidestream (SS) and mainstream (MS) CSC, the members of Project 6910 submitted duplicate SS and MS samples from five model cigarette types collected from an European design smoke collection chamber.³ All

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samples were dry impaction trap (DIT) collected and tested in strains TA98 and TA100 (+S9). An additional sample tested represented the residue left on the walls of the chamber after 2R1 SS collection.

The activities from all five model cigarette types indicated that MS CSCs were more active than the SS CSCs in both strains. All of the MS samples were more active in TA98 than in TA100. The activity of the 2R1 chamber SS sample was half the activity of the 2R1 IT SS counterpart.

The January progress report contained activity information on a set of 2R1 MS and SS fractions collected by several procedures.^{4,5} The results were difficult to interpret due to the unexpectedly high specific activity of the 2R1 SS sample. After analysis of the samples for water and nicotine, an accidental interchange of two samples was discovered and corrected. The 2R1 IT SS and an electrostatic precipitation collected SS sample had been mislabeled. The corrected results will be included in forthcoming 6910 and 6906 Annual Reports. As expected, the correctly identified 2R1 IT SS sample is now less active than the MS in both TA98 and TA100, which is in agreement with previous results.⁶

IV. REFERENCES

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3. McCoy, W. R.; Thompson, L. H. PM Notebook No. 7762/6-7, pp. 82-92; 93-104.
4. Thompson, L. H. Monthly Progress Report. Monthly progress report 83-013; 1983 February 15.
5. McCoy, W. R.; Thompson, L. H. PM Notebook No. 7762, pp. 80-81.
6. Burke, B. K. Monthly Progress Report. Monthly progress report 82-292; 1982 December 15.

Michael H. Tickle

nwp

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CHARGE NUMBER: 6908
PROGRAM TITLE: CHEMICAL STUDIES OF CONDENSATE
PROJECT LEADER: A. H. Warfield
WRITTEN BY: R. D. Kinser
PERIOD COVERED: January 1-February 28, 1983
DATE OF REPORT: March 8, 1983

PAH (R. Levins)

PAH III fractions and the strippings from the Florisil PR column used to generate the PAH III fractions from eight model cigarettes have been analyzed by GC with FID and NPD, and also submitted to Project 6906 for S/M assay (TA100 + S9). The strippings contain relatively polar compounds, possibly N-PAHs. Specific activities of the strippings were generally somewhat lower than those of the PAH-IIIs. Analysis of the GC data is in progress.

NITROSAMINE ANALYSIS (C. McKay, J. Millham, A. Warfield)

Efforts have been directed at improving the methodology employed in the analysis of volatile and nonvolatile nitrosamines. It was demonstrated that the ^{14}C labeled dimethylnitrosamine (NDMA) added to determine recovery does not interfere with NDMA quantitation. The steam bath used in sample concentration was replaced with a more precisely controlled water bath. Gas chromatographic conditions for nonvolatile nitrosamine determination were changed to diminish analysis time, and the chromatograph modified to enhance injection reproducibility. Studies were conducted to establish the optimum pyrolyzer temperature on the Model 610 TEA. N-nitrosodibutylamine was found to be a satisfactory internal standard for the analysis of volatile nitrosamines. Evaluation of the nonvolatile nitrosamine standards is now in progress.

BASE FRACTION ANALYSES (S. Tafur, R. Kinser, M. Zimmermann, C. Ellis)

Sephadex-LH20 fraction #7 from X6D3IM bases was separated by preparative LC. All the fractions generated were active in the S/M assay (Charge No. 6906). Chemical profiling is being used to guide isolation attempts. GC/MS profiles of seven fractions were generated. Harman and norharman are prominent components distributed throughout the fractions. Other compounds observed are in the molecular weight range 180-250 u and have mass spectra characteristic of aromaticity.

The nicotine level in the base fraction from LTF-5A + nicotine CSC was found to be 9.5%.

Levels of harman (H), norharman (NH), and 2-amino- α -carboline (2AC) were determined (by HPLC) in base fractions from 2R1 mainstream (MS) and sidestream (SS) smoke. Collection methods used were impaction trap (IT) (MS and SS), acid

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filled impaction trap (AIT) (MS and SS), and electrostatic precipitation (EPPT) (SS only). The SS levels of 2AC, H, and NH were less than MS levels for IT and AIT samples, but EPPT SS contained 2AC, H, and NH at levels comparable to those of AIT and IT MS.

TOXICITY OF PUPAL CELL CASING (S. Tafur, R. Levins, R. Kinser, M. Zimmermann)

Collaborative studies with Dr. D. Faustini to isolate the component(s) of pupal cell casings (PCC) responsible for the observed mortality of cigarette beetles (CB) fed mixtures of PCC and bright (Br) tobacco continued. Hexane extracts of PCC and Br loaded on Br at 5% and 1% levels resulted in CB mortality, but the Br hexane extract was of greater toxicity. GC and GC/MS analyses of the hexane extracts indicated that nicotine, neophytadiene, and waxes were the primary components. A CH_2Cl_2 :1.0 N HCl partition resulted in nicotine levels in both hexane extracts of <0.1%; the denicotinized extracts were not toxic suggesting that the toxicity observed for the hexane extract was due to nicotine. The PCC residue after hexane extraction was toxic, suggesting that the active agent was not extracted.

Insufficient amounts of material for feeding studies were obtained from CH_2Cl_2 extraction of the residue. Methanol extraction yielded a hygroscopic material which was loaded on Br tobacco at 15% levels for testing. Residues from hexane and methanol extracted PCC and Br tobacco are being tested at 30% loadings.

Robin D. Kinser

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